

STUDY ON THE RHEOLOGICAL PROPERTIES OF FAST-GROWING EUCALYPTUS

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ABSTRACT

To study the effect of retention and drainage aids on the fast-growing eucalyptus bleached kraft pulp (EBKP) suspensions, different concentrations of cationic polyacrylamide (CPAM) and cationic starch were dosed with EBKP suspensions and their effects on rheological properties were studied. The shear yield stress (τ_y) of softwood bleached kraft pulp (SBKP) and poplar alkaline peroxide mechanical pulp (PAPMP) were compared and evaluated for their potential applications. The results show that in the steady-state shear condition, the τ_y of EBKP is proportional to the pulp mass concentration (C_m), corresponding to the exponential relation $\tau_y = aC_m^b$. SBKP had the highest τ_y values, followed by EBKP and PAPMP. Adding CPAM and cationic starch to EBKP suspensions at low to moderate doses increased the τ_y , but once the levels reached a certain point, the τ_y values began to deteriorate. This research is crucial to achieve optimally and stabilize the downstream manufacturing process in papermaking.

KEYWORDS: CPAM, cationic starch, eucalyptus fiber liquid suspension, rheological properties, yield stress.

INTRODUCTION

Throughout the pulp and papermaking process, liquids and fibers are constantly in contact with one another, forming a fiber liquid suspension. The fiber liquid suspension used in the modern papermaking industry typically contains a range of polymers as additives to improve the properties of produced papers. The interactions between these substances constitute a complex fluid system (Goto et al. 1986, Seifu et al. 1994, Mackaplow and Shaqfeh 1996, Joung et al. 2001, 2002). The interaction of fibers and fluids is critical in the papermaking industry, as the consistency and stability of the pulp suspension affect the processing of pulp and the properties of the finished paper (Kuřna 2016, Brezániová 2016, Mosse 2012). Understanding the

dynamics of fluid-fiber interactions has become an important part of scientific research in the pulp and papermaking industries.

Within a fiber liquid suspension, fibers entangle and form network structures with mechanical strength, this strength provides the stability of network structures to resist external shear forces, which restricts free flow. The resistance to external shear force is referred as shear yield stress (τ_y). The τ_y is an important rheological parameter of pulp fiber suspensions and plays an important role in fluid research in the pulp and paper field, including pulp transportation, pulp mixing, pulp fluidization, paper forming, and computational fluidics simulation of pulp fiber suspension (Sha et al. 2015a, 2016a, Sha et al. 2020). Studying the τ_y of pulp suspensions can provide theoretical guidance for the pulp and paper field in both the production process and in the development and application of equipment (Sha et al. 2016b).

Retention and drainage aids are common process additives that act as flocculants and promote the aggregation of fillers, cellulosic fines particles, and fibers mainly by electrostatic interactions. The resulting flocs impart better drainability to the pulp suspension which allows for increasing the paper machine speed and reducing the expenditure of energy (Chi et al. 2007, Diab et al. 2015). Studying the effect of these additives on the pulp fiber liquid suspension can potentially lead to cost savings and material yield improvements in the papermaking process without changing normal machine operation (Qian 1997). Cationic polyacrylamide (CPAM) is a synthetic polymer, and its excellent control properties make it widely used in the pulp and paper industry (Hubbe et al. 2010, Cadotte et al. 2008, Swerin 1996). Cationic starch is a natural polysaccharide, and its low price and easy availability make it become one of the most commonly used retention and drainage aids so far (Diab et al. 2015). CPAM and cationic starch are two commonly used retention and drainage aids in the paper industry, so we selected to study their effects on yield stress.

The effect of polymer on interactions between fibers can be studied using a variety of methods. Test samples of paper are usually used in the industry to measure properties such as dry strength, wet strength, and the homogeneity of fiber distribution in the paper (Saito and Isogai 2007, Xu et al. 2007). However, such research is usually used empirical observations, with little insight into the pattern and mechanisms by which the polymer modifies interactions between fibers (Mosse 2012). Eucalyptus is recognized as a high-quality wood in the pulp industry. The cost of planting is low and economic benefits are extremely high (Ein-Mozaffari et al. 2005).

The actual transportation resistance of the pulp includes the frictional resistance between and inside the pulp flow layer and the pressure difference resistance of the fluid being sent. The internal friction of the fluid is the main reason for the actual conveying resistance of the pulp. The internal friction force of the pulp flow is related to the internal shear stress of the pulp, and this correlation is also affected by changes in the pulp concentration and flow velocity, and the yield stress is closely related to the shear stress (Sha 2015a). The purpose of this research is to study the effect of the concentration of CPAM and cationic starch, and the concentration of suspension mass on the kinetics of pulp flow and mixing in the pipeline. This study can provide a reference for the selection of suitable retention and drainage aids in the process of the papermaking industry, taking into account the yield stress of the fiber suspension on the premise

of ensuring retention and drainage effect, and providing a theoretical basis for the selective addition of CPAM and cationic starch.

The results can be used to provide a certain theoretical reference for the development of energy-saving equipment as well as reduce energy consumption and research on various sections in the pulp and papermaking process, such as pulp transportation, mixing, screening, paper machine flow, and sheet formation.

MATERIAL AND METHODS

Materials

Eucalyptus bleached kraft pulp (EBKP) was provided by Asia Symbol Pulp and Paper (Shandong, China). White granular powdered CPAM, with a molecular weight of 5 to 8 million and a charge density of approximately 10%, was provided by Xinxiang Ling Long Water Treatment Materials (Henan, China). The cationic starch powder was provided by Guangdong Hongxin Biotechnology (Guangdong, China).

Instruments

Brookfield RST soft solids tester rheometer (Brookfield, Middleboro, America) was used to characterize the yield stress of the pulp samples. ZQS-23 Valley beater (Shaanxi University, Xianyang, China) was used to disintegrate the pulp samples. A P95587 Schopper-Riegler freeness tester (Frank-PTI, Birkenau, Germany) was used to determine the refining degree of pulp samples. GBJ-A fiber breaker card (Changchun Yueming Small Testing Machine Co., Changchun, China) was used to organize the fibers. Fiber analysis was conducted using a MorFi Compact fiber morphology analyzer (Techpap, Grenoble, France). A halogen moisture analyzer (Precisa Gravimetrics AG, Dietikon, Switzerland) was used to determine the moisture content of the pulp.

Methods

Sample preparation

The moisture content of air-dried EBKP was measured by oven drying method. Weigh 25-35 g of pulp sheet in a clean and dried to constant weight weighing container, put it in the oven, open the lid of the container, and bake at $(105 \pm 2)^{\circ}\text{C}$ for more than 4 h. After the lid is closed, move the container into the desiccator, cool for 30 min and then weigh, then move the weighing container into the oven again, continue drying for 1 h, cool and weigh, and so on until the quality is constant. Moisture content x (%) is calculated according to the following formula:

$$x = \frac{m - m_1}{m} * 100\% \quad (1)$$

where: m - mass of pulp specimen before drying, g, m_1 - mass of pulp specimen after drying, g.

Two parallel measurements were carried out at the same time and the arithmetic mean was taken as the measurement result.

Calculate the required pulp sample and water consumption for pulping according to the moisture content of the pulp and the pulping concentration. Tear 360 g of absolute dry pulp sheet into small pulp pieces of 25 mm * 25 mm and soak them in 5L of water for more than 4 h. Add 12 L of water at temperature $(20 \pm 5)^\circ\text{C}$ to the Valley beater tank, slowly pour in the pulp and replenish water until the pulping concentration is 2%. Disintegrate the pulp until there are no small lumps and then add thallium to start pulping. The lever arm load of the Valley beater is 54 N as wood pulp is used. And the pulp was refined to a freeness degree of 45°SR . after the beating is finished, the pulp was centrifuged, placed in a sealed bag, and allowed to equilibrate for 24 h.

To measure the yield stress, 1L pulp suspension samples with mass consistencies (C_m) of 0.5%, 1%, 1.5%, 2.0%, 2.5%, 3.0%, 3.5%, and 4.0% were prepared. The cationic starch was gelatinized and the moisture content was measured before use, then prepared four concentration gradients of 1%, 1.5%, 2%, and 2.5%. CPAM solution was prepared with four concentration gradients of 0.01%, 0.3%, 0.6%, and 1% using a magnetic stirrer. Varying concentrations of either CPAM or cationic starch were added to the pulp suspension samples of varying consistencies. The yield stress values of the pulp suspensions were then measured using the RST rheometer.

Sample measurement

The yield stress was characterized using the shear stress gradient method of the RST rheometer. To reduce error, the average value of two measurements was used for analysis. An RST rheometer was used in the controlled shear stress (CSS) mode to gradually increase the shear stress from a small initial value to a certain value and to measure the shear strain versus shear stress curve of the pulp during this process. In the logarithmic coordinate system, the shear rate curve shows a clear stress plateau. The shear stress corresponding to the initial rotation of the rotor is the yield stress of the pulp fiber suspension.

RESULTS AND DISCUSSION

Relationship between C_m and yield stress for EBKP

The corresponding yield stress values for C_m samples of 0.5%, 1%, 1.5%, 2.0%, 2.5%, 3.0%, 3.5%, and 4.0% were 0 Pa, 2.8 Pa, 11.47 Pa, 20.64 Pa, 30.71 Pa, 58.93 Pa, 84.91 Pa, and 119.85 Pa, respectively. Fig. 1 depicts a graphic analysis of the yield stress values for EBKP at various consistencies.

The yield stress of EBKP suspensions increased as the C_m increased. The yield stress of EBKP suspensions showed a gradual increase in the 0.5% to 2.5% C_m range, and rapidly increased in the C_m range of 0.5% to 2.5%. The yield stress and C_m of pulp fiber suspension were assumed to satisfy the exponential function Eq. 1:

$$\tau_y = aC_m^b \quad (1)$$

where: τ_y is the yield stress (Pa), C_m is the mass concentration (%), and a and b are the constants related to fiber properties. Based on the experimental data, the values of a and b were obtained through nonlinear regression, as shown in Tab. 1.

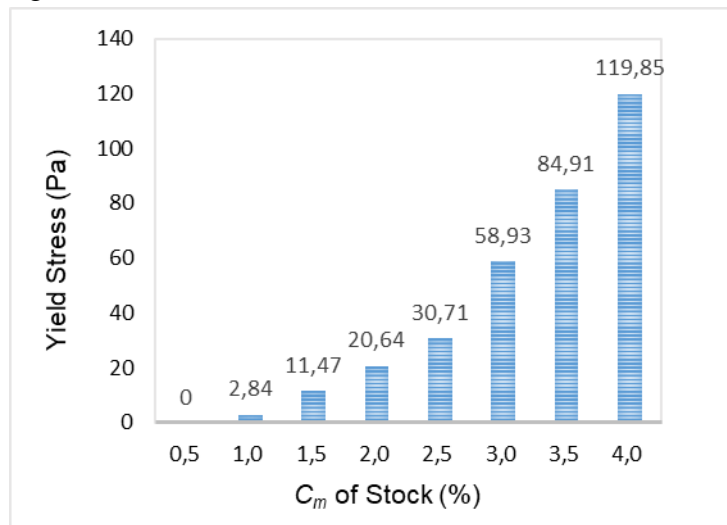


Fig. 1: The yield stress curve of EBKP suspensions at varying C_m values.

Tab. 1: Regression coefficient of yield stress index equation of EBKP suspension.

Pulp	$\tau_y = aC_m^b$		
	Parameter a	Parameter b	R^2
EBKP	3.197	2.616	0.997

The R^2 value is close to 1 indicating that Eq. 1 is an accurate model. The values of parameters a and b are within the ideal range of 1.18 to 24.5 and 1.25 to 3.02, respectively (Bennington et al. 1990). A modified version of Eq. 1 can be made into Eq. 2:

$$\tau_y = 3.197C_m^{2.616} \quad (2)$$

where: the a and b values have been replaced with the values from Tab. 1.

This index equation can be used to estimate the yield stress of EBKP suspensions with a specific C_m values. Higher C_m values resulted in higher yield stress values due to the higher number of fibers per unit volume. The increased fiber density allowed for more contact and entanglement, forming greater bond forces and interwoven grid structures (Zhang 2009).

Comparison of yield stress values for EBKP, softwood bleached kraft pulp (SBKP), and poplar alkaline peroxide mechanical pulp (PAPMP) suspensions

SBKP and PAPMP yield stress values increased as C_m increased, similar to that of EBKP (Chen et al. 2017, He et al. 2017). The measured results of EBKP were compared to the literature results for SBKP and PAPMP (Fig. 2).

Softwood pulp fibers are longer, bulkier, softer, and drain better than hardwood pulp fibers. However, due to the longer fiber length, softwood fibers tend to flocculate more during web

formation. Hardwood has a uniform size distribution with shorter fibers, and their size properties can affect drainage performance during the forming process.

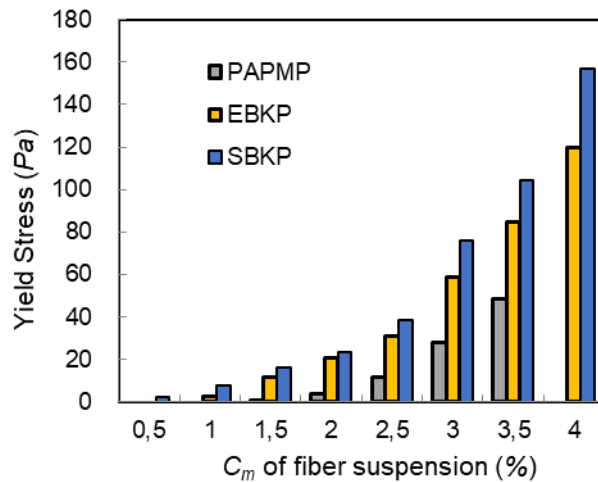


Fig. 2: Effect of C_m on the yield stress of PAPMP, EBKP, and SBKP suspensions.

Fig. 2 shows that the yield stress of both the PAPMP and SBKP suspensions increased nonlinearly with increased C_m . The yield stress of the SBKP was greater than that of PAPMP with a certain C_m , indicating that the degree of fiber-to-fiber interweaving in the SBKP suspension was much stronger than that of the PAPMP. This was attributed to the length of softwood fibers. A fiber analysis between two hardwood species, EBKP and PAPMP, was analyzed and compared (Tab. 2).

Tab. 2: Fiber properties of EBKP and PAPMP.

Pulp	Fiber length (number avg.) (mm)	Fiber length (weight avg.) (mm)	Degree of fiber curl (%)	Fine fiber content (%)
EBKP	0.662	0.762	11.8	22.5
PAPMP	0.532	0.648	8.8	56.6

The average fiber length of PAPMP was smaller than that of EBKP. The content of fine fiber with a length of less than 0.2 mm was higher in PAPMP. The degree of fiber curl was higher in the EBKP pulp. Because PAPMP was subjected to more cuts during refining, which resulted in shorter fibers. Fiber length and suspension C_m had a noticeable effect on the resulting yield stress. Longer fibers promoted fiber-to-fiber interweaving, which increased the binding force and thus increased the yield stress. The yield stress values of SBKP were higher than that of EBKP at a particular C_m , which were higher than that of PAPMP. At a C_m of 3.5%, the yield stress of SBKP and EBKP was 116.3% and 75.9% higher than the yield stress of PAPMP, respectively.

Effect of CPAM or cationic starch on the yield stress of EBKP suspension

Influence of CPAM on the yield stress of EBKP suspension

Varying dosages of CPAM can be used depending on the application. Higher amounts of CPAM resulted in a more severe aggregation of fibers within the pulp. Additionally, CPAM

overdosage can lead to a decrease in paper strength, due to the increased retention of fine fibers and filler. The dosed range of CPAM is generally from 0.01% to 1% (Hui et al. 2010). Therefore, this research selected four CPAM concentration levels of 0.01%, 0.3%, 0.6%, and 1% for yield stress testing. The selected concentration levels were added to EBKP suspensions with C_m values of 1.0%, 1.5%, 2.0%, 2.5%, 3.0%, 3.5%, and 4.0%. The resulting yield stress of the various EBKP suspensions after the addition of CPAM is shown in Fig. 3.

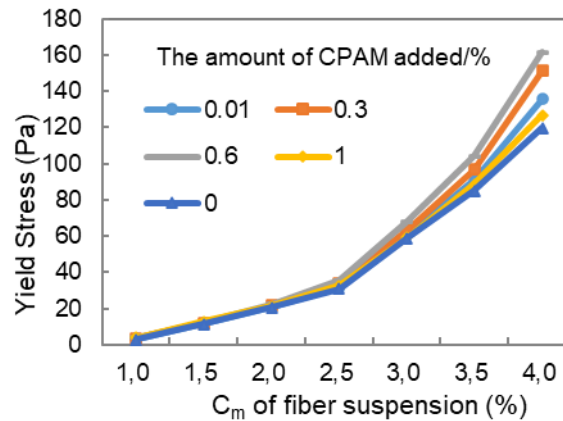


Fig. 3: The yield stress of EBKP suspensions with varying C_m after CPAM addition.

The results from Fig. 3 indicate that the addition of CPAM in moderate amounts had the greatest impact on the yield stress of EBKP suspensions. As the C_m of the suspension increased, a more apparent difference was seen among the varying dosages of CPAM. The CPAM addition levels of 0.6% and 0.3% resulted in the highest yield stress values, while CPAM addition at levels of 0.01% and 1% had less of an impact., the yield stress of the sample with C_m of 4.0% and 0.6% CPAM addition increased approximately 30% compared to that of the 4.0% C_m sample with no CPAM addition.

According to Eq. 1, the yield stress curve of EBKP suspensions depends on the C_m of the suspension. The values of a and b for CPAM dosages were obtained, as shown in Tab. 3. In Tab. 3, the R^2 values corresponding to different additive concentrations of CPAM were close to 1, which indicated that the equation was a good fit. The values of parameters a and b were within the ideal range of 1.18 to 24.5 and 1.25 to 3.02, respectively (Bennington et al. 1990).

Tab. 3: Regression coefficient of yield stress index equation of EBKP suspension after addition of CPAM.

CPAM Dosage (%)	$\tau_y = aC_m^b$		
	Parameter a	Parameter b	R^2
0.01	2.622	2.843	0.996
0.3	2.324	3.003	0.997
0.6	2.432	3.020	0.997
1	3.161	2.661	0.997

The concentration of the CPAM aqueous solution was small, so the yield stress influence of the solution itself on the results can be neglected. Additionally, CPAM is a linear chain polymer

compound, which is a very fine and soft structure compared to pulp fibers. Therefore, from the perspective of structure and molecular weight, the influence of CPAM structure on the yield stress values of the suspensions can be neglected. Consequently, it can be assumed that the electricity and charge density of CPAM were the main factors that affected the yield stress of the EBKP suspensions (Mosse 2012).

CPAM has a positive charge, its addition to the negatively charged fibers neutralizes their electrical properties. This causes flocculation of fiber fines and promotes interweaving among the fibers, and forms a myriad of tiny fiber clusters. Using a rheometer rotor to disperse these fiber clusters requires more force, which means increasing the yield stress of the fiber suspension (Xu et al. 2001). However, when the CPAM concentration increased to a certain amount, the CPAM acted as a wrapping agent, which caused the fiber flocculation groups to become smooth. In this case, the CPAM created a dispersion effect and reduced the yield stress. However, suspensions with over-dosed CPAM still exhibited higher yield stress values compared to suspensions with no CPAM addition.

Effect of cationic starch addition on the yield stress of EBKP suspension

Cationic starch is an important and widely used chemical additive in the paper industry. The dosage of cationic starch as a retention aid is generally between 0.03 and 0.07 (Wei and Zhang 2012). The amount of cationic starch used depends on the paper machine configuration and the grade of paper being produced. In general, smaller paper machines often use unary or binary additives, while larger machines use a relatively complex variety of retention aids (Hubbe and Nanko 2010). This disparity makes it difficult to find the optimum dosage for each element of a retention aid, which needs to be explored and summed up through specific production experience.

In this research, 1.0%, 1.5%, 2.0%, and 2.5% of the cationic starch additive concentration were set to study the yield stress values of EBKP suspensions at C_m values of 1.0%, 1.5%, 2.0%, 2.5%, 3.0%, 3.5%, and 4.0%. The yield stress values of EBKP suspensions with different concentrations of cationic starch were plotted against the different C_m values, as shown in Fig. 4.

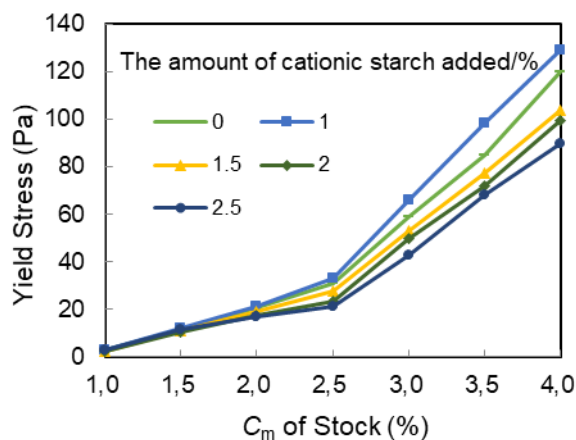


Fig. 4: Change in yield stress for EBKP suspensions of varying C_m with cationic starch addition.

As shown in Fig. 4, the yield stress of EBKP suspensions increased nonlinearly with increasing C_m suspension. Assuming that Eq. 1 is satisfied, the values of a and b were obtained from a nonlinear regression analysis. The results are shown in Tab. 4, the correlation coefficient R^2 was close to 1, which indicated that the equation was a good fit. The values of parameters a and b were within the ideal range of 1.18 to 24.5 and 1.25 to 3.02, respectively (Bennington et al. 1990).

Tab. 4: Regression coefficient of yield stress index equation of EBKP suspension after addition of cationic starch.

Cationic starch dosage (%)	$\tau_y = aC_m^b$		
	Parameter a	Parameter b	R^2
1.0	3.653	2.587	0.992
1.5	3.299	2.496	0.995
2.0	2.650	2.620	0.991
2.5	2.552	2.575	0.987

As shown in Fig. 3, when the cationic starch content was 1%, the yield stress of the EBKP suspension increased by approximately 7.6%. At cationic starch dosages of 1.5%, 2%, and 2.5%, the yield stress decreased. As the C_m of the suspension increased, the yield stress deviation among the samples became more pronounced. At the highest dosage level, the yield stress is approximately 25.4% less than the sample with no cationic starch addition. Similar to the CPAM, the electrical properties and charge density of the cationic starch were the main factors that affected the yield stress of the pulp suspension.

Cationic starches with positive charge can bind tightly to negatively charged fibrils and fillers (Qin 2002). At a low dosage (1%), cationic starches were mainly combined with the pulp fibers through charge neutralization in the fiber suspension system. The cationic starch acted as an intermediary, forming a larger floc with a mixture of long fibers and fine fibers and increasing the yield stress. A fiber surface area can only adsorb a certain amount of cationic starch (Marton and Terezia 1976). As the fiber adsorbs a given amount of cationic starch, the adsorption points on the fiber are reduced. If cationic starch addition continues past the certain dosage level, it will act as a dispersant and separates the large fiber flocs to form many smaller flocs with fine fibers. Less shear stress is required to disperse the fine fiber flocs, thus decreasing yield stress.

Comparison of the effects of CPAM and cationic starch on the yield stress of EBKP

Both the CPAM and cationic starch act as retention aids through charge neutralization, fiber-bridging, and patch mechanism, but their chemical structures and molecular weights are different. Additionally, CPAM and cationic starch have varying contents of cationic monomers, which resulted in a varying number of adsorption sites bound to fiber surfaces. As a result, the fiber and filler flocs formed are different, which affects the retention properties of the additives. Comparisons between CPAM and cationic starch can be made from the analysis of Figs. 3 and 4. When dosed at low to moderate levels, both additives were shown to increase the yield stress of the fiber suspension. The retention aid additives created flocculation within the suspension, created interweaving of fibers, and thus increased yield stress. However,

as the additive dosage increased, the adsorption points of the pulp fibers began to decrease, leaving fine fibers as the sub-centers for the formation of network flocs. These fine fiber flocs had a rolling and sliding effect, which caused the yield stress of the suspension to gradually decrease. Cationic starch provides good adhesion to fibers and can be easily dispersed. Therefore, when added to a fiber suspension, cationic starch tends to form more small fiber flocs, which led to a noticeable reduction in the yield stress of the suspension compared to CPAM.

CONCLUSIONS

This study focused on the effects of CPAM and cationic starch on the τ_y of fast-growing eucalyptus fiber liquid suspension with different concentrations. The physical model of adding CPAM or cationic starch to eucalyptus pulp was obtained according to the regression equation, and the equations are well-fitted by R^2 verification. Besides, the τ_y of long-fiber softwood pulp and short-fiber hardwood pulp within a certain fiber mass concentration range are compared. The following conclusions are obtained: (1) The τ_y of EBKP nonlinearly increased with increasing pulp consistency. The τ_y was 0 Pa at a C_m of 0.5%, and increased to 19.85 Pa when the C_m was 4%. The relationship between τ_y and C_m was satisfied by Eq. 2 when C_m values were greater than 0.5%. (2) At a given pulp consistency, the τ_y values of SBKP were higher than that of EBKP, and the τ_y values of EBKP were higher than that of PAPMP. The τ_y of pulp suspensions seemed largely dependent on fiber length, with longer fibers exhibiting larger τ_y values. When the pulp consistency was 3.5%, SBKP pulp τ_y was 116.3% higher than PAPMP pulp τ_y , and EBKP pulp τ_y was 75.9% higher than PAPMP pulp τ_y . (3) A moderate CPAM dosage level of 0.6% yielded the highest τ_y among the samples. The highest CPAM dosage of 1% yielded the lowest τ_y among the dosed samples, but it was still higher than that of the EBKP with no CPAM. The effects of CPAM on τ_y were easily seen in the suspensions with $C_m = 4.0\%$. After the addition of CPAM, the pulp suspension still satisfied Eq. 1, but with altered values for constants a and b depending on the dosage level. (4) The addition of cationic starch had mixed effects on the τ_y values of EBKP suspensions. The lowest dose of cationic starch (1.0%) was the only sample to increase the τ_y of EBKP suspensions. As cationic starch was dosed in quantities, the τ_y of EBKP decreased. The effects of cationic starch on τ_y were most apparent at $C_m = 4.0\%$. After the addition of cationic starch, the pulp suspension still satisfied Eq. 1 but with altered values for a and b depending on the dosage level. (5) The addition of CPAM in the normal production process did not contribute to reducing the τ_y of the fiber suspension, when the amount of cationic starch was in the range of 1.5% to 2.5%, it had a noticeable contribution to reducing the τ_y of the pulp fiber suspension, when the pulp concentration was higher, the effect was more obvious. Cationic starch should be preferred retention and drainage aids over CPAM by the papermaking industry.

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