Paper III

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Characterization of chemical pulp fines

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ABSTRACT: This study presents a number of new methods for characterizing chemical pulp fines. The methods are used to measure the dewatering rate, settling rate, and viscosity of a fines suspension, which are closely related to the specific surface area of fines. The dewatering rate of the fines suspension measured with a gravimetric water retention meter that used pressure filtration coincided with the results obtained with a conventional method for measuring the drainage time in a sheet mold. The settling rate measured with a Turbiscan device, which is equipped with an optical head for scanning the sample automatically, correlated with the results of conventional visual observation. The Brookfield viscosity of the fines suspension may be dependent on the degree of hydration of the fines network. All of these new methods were found to be simple, fast, and accurate for measurement. The finer the fines fractions, the more it contributed to slowing down the settling and increasing the viscosity of the fines suspension.

Application: This study presents several simple methods for characterizing chemical pulp fines, allowing better control of fines quality.

Chemical pulp fines are an important component in papermaking furnish. They can significantly affect the mechanical and optical properties of paper and the drainage properties of pulp [1-2]. Characterizing the fines will therefore allow a better understanding of the role of fines and better control the papermaking process and the properties of paper. Several attempts have been made to characterize fines in terms of size [1-10], shape [6-8,10-11], swelling ability [1,8-9], drainage properties [1-3,12], specific surface area [2-3,13-15], and chemical composition [6-9,16].

To measure these characteristics, fines are typically prepared by prolonged refining, and then separated and collected using a 100-mesh or 200-mesh screen of a Bauer-McNett classifier. The classifier allows dissolved and colloidal substances to be washed away [17]. Sometimes fines are collected as sediment after several days. This is a rather long and laborious procedure. However, fines can be easily prepared with an ultra-fine friction grinder [18]. Screening with a Bauer-McNett classifier to collect fines may not even be required, because 100% fines can be produced by using an extremely small gap clearance in the grinder.

Chemical pulp fines retard dewatering of the pulp suspension due to the high water holding capacity of fines [9,19]. In the conventional method for characterizing the role of fines in dewatering, a proportion of fines is added to the fiber furnish, and then only the drainage time of the whole furnish is measured in a standard sheet mold. Unless an optimum amount of fines is added to the fiber furnish, the drainage becomes too slow, and some fines are lost during the test. To solve this problem, a gravimetric water retention meter [20], which is often used to measure the water retention of coating color, can be used to measure the amount of water drained from the fines suspension in a given time and at a given external pressure. This fast and simple method is based on pressure filtration, with the water draining through a filter medium under external pressure [20].

The specific surface of fines can be considered an important characteristic of dewatering and bonding ability. This can be measured indirectly from the settling rate, because the settling rate is strongly dependent on the specific surface area [10]. A conventional visual technique for observing the interface of the sediment-water as a function of time can be used, but it is difficult to read the interface accurately because of the fast settling and uneven interface used in this method. An automatic detection system to read the interface would be beneficial for measuring the settling rate. The Turbiscan MA 2000 apparatus [21] with a detecting head (composed of a near-infrared light source and two synchronous detectors) scans the sample automatically at a given time and could be an efficient tool for measuring the settling of a fines suspension accurately.

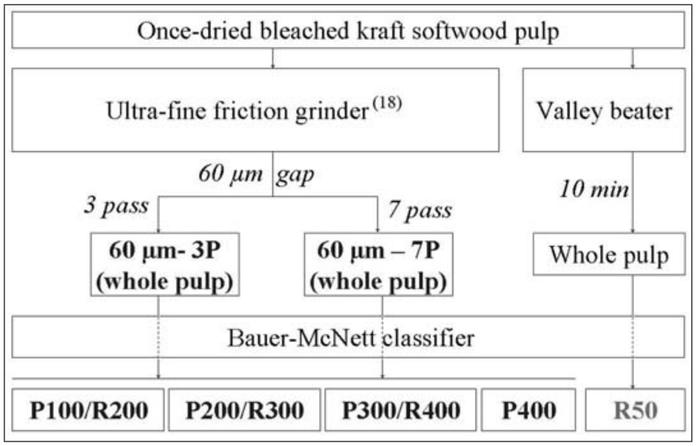
A fines suspension is composed of heterogeneous fines particles in water. The suspension exhibits different rheological characteristics depending on the degree of interaction between the fines particles and on their hydration. Although viscosity measurement, which is simple and easy, has not been used for fines characterization, it may provide useful information.

The characteristics of fines, such as the dewatering rate, settling rate, and viscosity of the fines suspension, are closely related to the specific surface area of fines. The objective of this study was to present several simple methods, as mentioned above, for characterizing chemical pulp fines, which would allow the quality of fines to be controlled more effectively.

EXPERIMENTAL

Fines preparation

Once-dried bleached kraft softwood (a mixture of Scots pine and Norway spruce) pulp was used to produce fines with an ultra-fine friction grinder (super masscolloider®, Masuko Sangyo Co. Ltd., Japan) [18]. The pulp samples were ground into fines by circulating them three (3P) and seven (7P) times through a gap of 60 µm. The pulps were then fractionated in a Bauer-McNett classifier using 100-mesh, 200-mesh, 300-mesh and 400-mesh screens according to the modified SCAN-M6:69, and the ground pulp (60 µm-3P and 60 µm-7P) and its sub-fractions (P100/R200, P200/R300, P300/R400 & P400) were



1. Fines preparation.

used for the characterization. The fines preparation procedure is summarized in **Fig. 1**.

The pulp fractions were beaten separately for 10 min in a Valley beater (ISO 5264-1:1979). A long fraction was then collected using a 50-mesh screen in a Bauer-McNett classifier. This fraction was mixed with fines and its drainage properties determined in a standard sheet mold.

The fines contents (P100) of the two pulps ground in the grinder were 87% and 97% (**Table I**). The grinder was a very effective tool for producing pulp fines.

The consistency of the fines suspension was adjusted for the characterization (**Table II**).

Fines characterization

The mean size (equivalent volume diameter) of the whole fines and their sub-fractions was measured using a Coulter Multisizer II (Coulter Electronics Limited, England).

Light microscopic imaging of the fines fractions was carried out using a DM LAM microscope (Leica, Germany). Two different modes were used, phase contrast and bright field with polarized light.

The dewatering rate of the fines suspension was measured using a gravimetric water retention meter (DT Paper Science Oy, Finland) [20]. Blotting paper was used as absorbing paper. A membrane filter having a pore size of 0.22 µm (Millipore, USA). 10 mL of fines suspension was used for the measurement. The external pressure varied from 0.2 bar to 0.4 bar, and the pressurized time from 10 sec to 50 sec. Water removed from the fines suspension through the filter is absorbed by the blotting paper at a given pressure and within a given time. The difference in the weight of the blotting paper before and after water absorption is then calculated in grams per square meter (g/m^2). Further details of the operation of the gravimetric water retention meter can be found in an earlier publication [20]. To compare the results produced by the gravimetric water retention meter with those obtained with the conventional method, the drainage time was measured separately using a standard sheet mold according to SCAN-M9:76. We used 30% of the fines suspensions (60 µm-3P and 60 µm-7P), each mixed with the fiber fraction (R50), to determine the drainage time in a sheet mold.

The settling rate of the fines was measured with a Turbiscan MA 2000 device (Formulaction, France) [21] in which the

Fraction	60 µm-3P	60 µm-7P
Long fiber fraction (R30)	1 %	0 %
Middle fiber fraction (P30/R100)	12 %	3 %
Fines (P100)	87 %	97 %

I. Comparison of two different pulps beaten in ultra-fine friction grinder.

sample is inserted into a cylindrical glass tube (diameter = 12 mm, height = 140 mm). An optical head using a near-infrared light source (850 nm) vertically scans the length of the sample (65) mm, recording the transmitted and back-scattered intensities. The settling rate of the fines fractions was calculated from transmitted intensities of 12-time scans. We then compared the results from the conventional method [10]—observing the interface of the sediment and water with the naked eye for a given time—with the results obtained with the Turbiscan method.

The apparent viscosity of the fines suspension was measured using a Brookfield DV-II viscometer (Brookfield Engineering Labs., Inc., USA) according to SCAN-P 50:84. We used spindle No.3 and 100 rpm in taking the measurement. The temperature of all fines suspensions was 22°C.

RESULTS AND DISCUSSION

The mean size of each fraction of fines collected using the Bauer-McNett classifier was measured with a Coulter multisizer (**Table III**). The mean diameter provides a good representation of the corresponding fraction of fines. The heterogeneous structure of fines makes it is difficult to measure the actual average length of fines (**Fig. 2**). The complicated structure of fines shown on the left was revealed using the contrast mode of the light microscope. The enlarged and detailed structure, containing plenty of thread-like fibrils shown on the right was revealed using a bright field with polarized light. Fines are not seen as an individual fraction having a certain length or shape, but as an entangled network of fibrils.

Figure 3 shows the dewatering rate of two fines suspensions, 60 µm-3P and 60 µm-7P, containing 87% and 97% of fines (P100), respectively, measured with a gravimetric water retention meter. The difference in the amount of water removed at the lower external pressure appears to be similar for the two suspensions, but the difference grows with higher external pressure and duration of pressurization. It is therefore preferable to apply the higher external pressure for the measurement. From the suspension containing more fines, less water is drained at the higher external pressure as a function of time. For the purpose of comparison, the drainage times of the two suspensions were measured in a sheet mold. The drainage times of the 60 µm-3P and 60 µm-7P suspensions were 14 sec and 30 sec, respectively, which coincides with the results obtained with the gravimetric water retention meter. The water retention meter offers several advantages over the conventional method:

- It can measure dynamic changes in dewatering
- It only uses fines for measurement
- It is a simple and fast method.

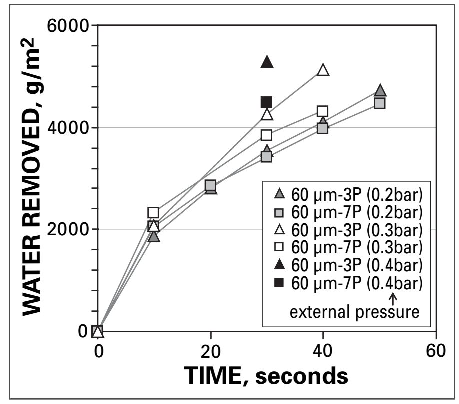
Figure 4 shows the settling rate measured with the conventional and the Turbiscan methods. The measurements correlate

Measurement	Consistency, %
Dewatering (gravimetric water retention meter)	1.00
Settling rate (Turbiscan MA 2000 & conventional)	0.03
Viscosity (Brookfield)	1.00

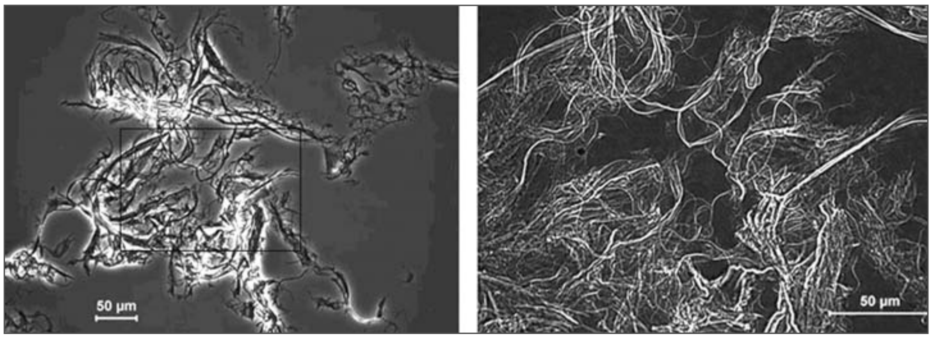
II. Pulp consistency for the measurements.

Fraction	Mean diameter, µm
P100/R200	16.6
P200/R300	11.4
P300/R400	11.1
P400	8.5
Whole pulp	9.8

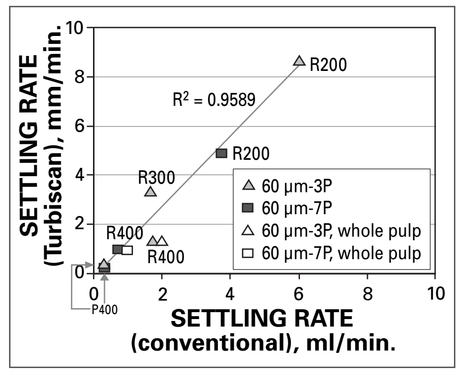
III. Mean diameter of different fines fractions, 60 µm-7P.



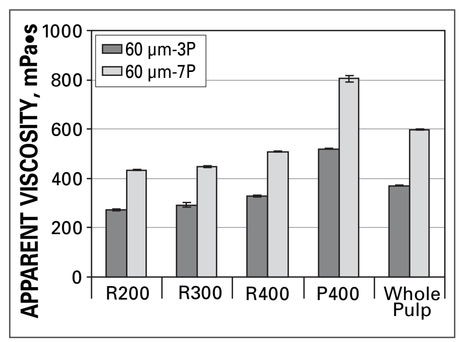
3. Dewatering rate of fines suspensions containing different amounts of fines.



2. Microscopic images of fines (P100 fraction), 60 µm-7P. Phase contrast mode (left) and bright field with polarized light mode (right).



4. Settling rate of fines fractions measured with two different techniques.



5. Apparent viscosity of fines fractions. Brookfield viscosity at 100 rpm with spindle No. 3.

well with each other. As the size of the fines fraction decreases, the settling rate of the fraction also decreases, as expected. The Turbiscan method offers certain advantages over conventional visual observation. First, the result is very accurate, because the detection head, composed of a light source, automatically detects the interface of the sediment and water in the fines suspension. Second, it is an easy and fast method.

Figure 5 shows the Brookfield viscosity of a fines suspension containing different fines fractions. According to the experiments, a greater force is required to rotate the spindle in the finer fines suspension, thus indicating higher viscosity. Because the finer fines contain more bound water, they may behave like a strong gel. Fibrils, as shown in Fig. 2, form a three-dimensional network in water, which behaves like a gel due to the high amount of bound water [9,19,22,23]. The whole fines suspension, 60 µm-7P, containing more fines, has a higher viscosity than the 60 µm-3P suspension. Viscosity is simple and easy to measure. The fraction passing through a 400-mesh screen has a much higher viscosity compared to the other fractions, R200, R300 and R400.

CONCLUSIONS

Chemical pulp fines produced with an ultra-fine friction grinder were characterized using several simple and new techniques. The following conclusions can be drawn:

- The dewatering rate of the fines suspension obtained using a gravimetric water retention meter coincided with the results obtained by measuring the drainage time in a standard sheet mold. The gravimetric water retention meter offers advantages over the conventional method. It can measure dynamic changes in dewatering; it only used fines for the measurement; and it is an easy and simple method.
- The settling rate measured with a Turbiscan MA 2000 device, which is fast, accurate, and easy to operate, correlated with the results of conventional visual observation of the interface of the sediment-water as a function of time.
- The viscosity of the fines suspension measured with a Brookfield viscometer, which also is simple and easy to measure, might provide information on the degree of hydration of the fines network.
- The finer the fines fraction, the more it contributed to slowing down the settling and to increasing the viscosity of the fines suspension. **TJ**

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INSIGHTS FROM THE AUTHORS

It is well known that pulp fines play an important role in the paper-making process and for the properties of paper. Even a comparatively small amount of fines can influence paper properties significantly. Better knowledge of the characteristics of fines would be beneficial for controlling the quality of fines and for using the full potential of fibrous materials. Therefore, an attempt was made to find a simple, accurate, and fast method to characterize fines.

The most difficult aspect in the early stage of the research was how to produce a large amount of fines efficiently for use in measurements. An ultra-fine friction grinder was found to be an effective means to produce fines without prolonged refining. Adjusting the variables of the grinder, such as a gap clearance, rotating speed, and the number of pulp recirculations through the gap, makes it possible to control the fibrillation of fibers and the production of fines.

The gravimetric water retention meter and the Brookfield viscometer (which is typically used for determining the characteristics of coating color) were found to be excellent tools for characterizing fines suspensions. The Turbiscan method, because of its accurate and simple measurement procedure, also can be used to replace the conventional visual method for measuring the settling behavior of fines particles.

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