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The importance of cellulosic fines relative to the dewatering rates of fiber suspensions

Hao Chen, Andrew Park,† John A. Heitmann, and Martin A. Hubbe*
Dept. of Forest Biomaterials, North Carolina State Univ., Box 8005, Raleigh, NC, USA 27695-8005; * hubbe@ncsu.edu
Footnote: † Clemson University, 200 Orange Court, Clemson, SC 29631; ampark@clemson.edu

Abstract
When cellulosic fines are present in significant amounts they can have a dominant influence on dewatering. Pulp suspensions drain rapidly if the fines have been removed. In this study, the dependency of gravity dewatering rates on the level and properties of cellulosic fine matter was quantified. Bleached hardwood kraft pulp was used as a source of primary fines (collected before refining) and secondary fines (collected after refining of fines-free fiber suspensions). Fractions of fine matter also were obtained from chemithermomechanical (CTMP) pulp. Size distributions of these fines were characterized using a laser diffraction method. Results were explainable by a mechanism in which unattached fines are able to move relative to adjacent fibers during the dewatering and consolidation of a mat of fibers. Due to such movement, fines end up in locations where they plug drainage channels in the mat. The contribution of the fines to dewatering increased in inverse proportion to particle size and with increasing surface area, as calculated from the light scattering analysis.

1. Introduction

Fine cellulosic particles, i.e. "fines," can play a pivotal role in the manufacture of paper. On the one hand, fines can help to fill in the spaces between fibers in a sheet of paper. This effect typically results in a denser, stronger, and more uniform product.[1-3] On the other hand, high levels of fines can make it more difficult to remove water from the wet sheet of paper, as it is being formed .[4-12] The latter effect can slow down the manufacturing process or increase the amount of energy that is required in order to dry the paper.

Past studies have revealed dramatic differences in the sizes and shapes of cellulosic fine material derived from different sources and resulting from different levels of mechanical treatment.[4, 7, 13-14] The pioneering work of Brecht and Klemm[4] focused on quality aspects of the fines component in groundwood pulps. These authors described some of the fines as granular ("Mehlstoff"), whereas other fines in groundwood are mucilaginous, consisting of slender fibrils ("Schleimstoff"). More recent research has found similar contrasts between the fine components obtained by screening of thermomechanical pulp (TMP) samples.[13-14]

Studies of various kraft pulps have likewise revealed two contrasting types of cellulosic fines.[2-3] So-called "primary fines" consist of ray parenchyma cells and other non-fibrous materials that are liberated from the wood by chemical pulping. These fines, though somewhat diverse and species-dependent, can generally be described as "short,"

and their length-to-thickness ratio is usually less than 5. By contrast, "secondary fines" from kraft pulps are produced by refining action, which causes some of the primary wall or secondary wall layers to delaminate and become detached from the fibers, especially at high levels of refining. Such secondary fines tend to be slender and flexible.

Previous studies in our research group showed significant differences in the ability of primary vs. secondary fines from bleached hardwood kraft pulp to reduce the rate of water release from fiber suspensions.[9,12] These results left some mechanistic questions unresolved, since primary and secondary fines differ from each other both with respect to size and shape. One objective of the present work was to find further evidence related to these two variables. Accordingly, this study employs a wider range of cellulosic fines, together with more detailed analysis of particle size distributions and microscopic imaging. In addition to primary and secondary fines from bleached kraft pulp, a sample of chemithermomechanical pulp (CTMP) was fractionated to obtain various size ranges of fines. All of the fines were evaluated in the presence of fibers taken from a master batch of fines-free repulped copy paper. The overall aim of the work has been to show how attributes of fiber fines, as well as their proportion in the furnish, affect the rate of release of water from fiber suspensions during a gravity dewatering test.

An additional factor that was considered was the water-swollen nature of cellulosic fines, in comparison to the fibers. Water retention value tests,[15-17] based on centrifugal removal of water between the fibers, have been used to estimate the amount of water held within the cell walls of kraft fibers. More recently, Kang and Paulapuro pioneered the use of an alternative method to obtain similar information about fiber fines.[18] Their method was an adaptation of a water retention method that had been developed for evaluation of water retention in coating color formulations.[19] In the present study we have made a further modification to the method in order to be able to express the results as "grams water per gram of dry fiber," as is the standard form for reporting the corresponding results for water retention values of pulp samples.[15-16]

2. Materials and Methods

2.1. Preparation of default fibers. Except where noted otherwise, the (fines-free) fibers used throughout the study were prepared by dispersing torn pieces of 20lb., 84 brightness, 35% recycled content xerographic copy paper and then collecting the fiber fraction using a Bauer-McNett classifier with a 100-mesh screen (see TAPPI Method T233). The classifier was run for 15 minutes during each batch treatment, and the filtrate passing through the screen was discarded.

2.2. Preparation of hardwood bleached kraft pulp fines. The kraft pulp fines were prepared from dry-lap hardwood bleached kraft pulp from a local paper mill. The wood species distribution was not evaluated. The fibers were bleached by a conventional approach, including chorine dioxide. The pulp sheets were first torn into *ca.* 5 cm square pieces, soaked for a few minutes in tap water, then disintegrated in tap water according to TAPPI Method T205. Two different kinds of fines were collected. First, a set of "primary fines" was collected by use of a 100-mesh screen in the final chamber of a Bauer-McNett classifier (see TAPPI method T233). The water passing through the 100-mesh screen was collected in a 50-gallon drum, which was allowed to stand overnight

without stirring. The supernatant solution was then siphoned off. The settling and siphoning operation was repeated with a smaller volume for another overnight period in order to achieve the needed solids content of 1% or more.

Secondary fines were prepared by taking the material retained by the 100-mesh screen (*i.e.* fines-free bleached hardwood kraft pulp) and refining it extensively with a laboratory Hollander beater (TAPPI Method T200) to a Canadian Standard Freeness (TAPPI Method T227) of 100 ml. The mixture was then placed in the final chamber of the Bauer-McNett classifer, only this time a 200-mesh screen was used. The finer screen was used based on preliminary observations that the high level of refining rendered the fibers so flexible that they were able to pass through a 100-mesh screen. The filtrate was collected in a 50-gallon drum and allowed to settle for two days before the supernatant solution was carefully siphoned off. As in the previous case, the settling and siphoning process was repeated with smaller volumes, as needed, until a suitable solids content had been obtained.

2.3. Preparation of chemithermomechanical pulp fines. The CTMP fibers were type Q120.60 from Quesnel River Pulp Co. These fibers were provided with a Canadian Standard freeness of 120 ml and a brightness of about 60%. The fibers were disintegrated in tap water according to TAPPI Method T205. The Bauer-McNett apparatus was used to prepare the following fractions: R100 (passing a 48-mesh screen, but retained on the 100-mesh screen), R150 (passing the 100-mesh screen but retained on the 150-mesh screen), R200 (passing the 150-mesh screen but retained on the 200-mesh screen), and P200 (passing through the 200 mesh screen and collected by over-night sedimentation in a barrel).

2.4. Microscopy. Optical microscopy was carried out with an Olympus BH2 microscope, using either a 10X or 20X objective. Images were recorded with a TV camera. Dimensions were calibrated with a standard length. Samples were placed onto glass slides at various dilutions, with the goal of being able to simultaneously view 10-100 particles in one field, while at the same time avoiding excessive contact or overlap among the particles.

2.5. Particle size analysis. The apparent particle size distributions of cellulosic fine matter were determined with a Horiba LA-300 laser diffraction device. This apparatus measures the intensity of laser light that is scattered at various fixed angles relative to the transmitted light through a dilute suspension. Concentrations of fines samples in water were adjusted so that the percent transmittance of light was within the specified range. Default conditions for ultrasonication (before the measurement) and recirculation of the suspension were used. Particle size distributions were weighted by the software on an effective surface area basis.

2.6. Water release vs. time. Gravity drainage tests were conducted with a portable modified Schopper-Riegler test device assembled and donated by Buckman Laboratories International, Inc. The essential parts of this apparatus match those in the standard Schopper-Riegler test specification (ISO 5267/1, BS 6035/1 and SCAN C19), except that a single, large opening is provided at the base of the collection cone for the

filtrate. Also, a 100-mesh stainless steel screen was used. Suspensions were prepared with sufficient Na_2SO_4 so that the solution conductivity was 1000 µS/cm, and dewatering tests were done at room temperature (*ca.* 24 ^oC). Cellulosic suspensions were prepared at a solids content of 0.5%; in other words, the total amount of cellulosic material was kept constant, even if the fines level was varied. The suspensions were stirred with an impeller at a fixed, moderate speed for 30 seconds (or another fixed period in certain sets of tests) before adding the suspension to the top part of the test device. The amount of filtrate was determined with an electronic balance as a function of time after releasing the suspension and allowing it to impinge onto the screen.

2.7. Filtrate turbidity. Turbidity tests were carried out to obtain information related to the retention of cellulosic fines in the fiber mat. During previous work in our lab [9] a good correlation had been obtained between turbidity and the concentration of fines suspended in aqueous solution. The turbidity values were obtained with a DRT-15CE turbidimeter from HF Scientific (Ft. Meyers, Florida, USA). Sample cuvettes were individually inverted about 1-2 seconds before each reading to ensure an even suspension of the fines.

2.8. Water retention. The relative abilities of the fiber and the fines fractions of the bleached hardwood kraft pulp to retain water were evaluated by a modification of the method used by Kang and Paulapuro.[18] The tests were carried out with a Gravimetric Water Retention Meter (design by DT Paper Science Oy, Finland; model AA-GWR), which is usually used to measure the water retention in coating color.[19] The added samples were at 0.5% solids and 5 mL sample volume, for a filter area of 8 cm^2. Polycarbonate 5 µM membrane filters were used, backed by four layers of blotter paper to absorb the expelled water. The applied air pressure was set at 68.9 kPa (10 psi), and the pressure was applied for periods of 5, 20, 30, or 60 minutes. At the end of the selected time, the air pressure was discontinued, and the mass of the damp cellulosic material was determined (damp mass). The cellulosic material was dried at 105 ^oC for 60 minutes, then weighed again (to determine dry mass). The water retention was defined according to the following formula,

$$WR = [\text{wet mass} - \text{dry mass}] / (\text{dry mass}) \quad (1)$$

3. Results and Discussion

3.1. Cellulosic fines from bleached hardwood kraft pulp. The primary and secondary cellulosic fines, obtained from hardwood bleached kraft pulp, were characterized by light microscopy and laser diffraction particle size analysis. As shown in Fig. 1, the primary fines (the "pass" fraction through a 100-mesh screen, from a suspension of unrefined pulp) consisted mainly of relatively short objects, as would be expected due to the presence of parenchyma cells, parts of vessels, and other small features in delignified hardwood kraft pulp. Though some of the observed objects had length-to-thickness ratios as high as about ten, most of the particles could be described as "blocky" or "rectangular," rather than "fibrillar."

Figure 2 shows a representative image of secondary fines, which were obtained by refining the "retained" fraction just described, *i.e*. the bleached hardwood kraft fibers. After refining the pulp to a Canadian Standard freeness of 100 ml, the suspension was classified with a 200 mesh screen. The finer screen was employed because preliminary tests had shown that the 100 mesh screen did not effectively exclude refined fibers from the filtrate, presumably because the extensive refining had greatly increased fiber flexibility. Although some of the objects appearing in Fig. 2 may have the same origin as those in Fig. 1, *i.e.* "primary fines," the image also shows a lot of highly fibrillated, slender, and almost transparent material. It would be expected that the refining of kraft fibers will cause some delamination of the primary and S1 layers of the fiber, and maybe the outer portion of the S2 sublayer. Indeed, images of fibers (*e.g.* Fig. 3) revealed extensive fibrillation at the fiber surfaces.

Particle size distributions of the kraft pulp fines, based on laser light diffraction, are given in Fig. 4. As shown, the primary fines had a bimodal distribution. By contrast, the secondary fines were unimodal, and the distribution did not reach values as high as in the case of the primary fines. The difference in effective maximum sizes is consistent with the use of different size screens for the respective fractionation steps. In addition, because the laser diffraction results are affected by both the length and the thickness of the observed objects,[20] very thin, fibrillar material in the secondary fines would be expected to show up as smaller objects, compared to primary fines of equal length.

Results of water retention tests, based on the pressurized air method (see section 2.8) are shown in Fig. 5. As shown, the water retention decreased during the first 30 minutes of pressure application, but essentially the same results were obtained for pressure application times of 30 and 60 minutes. Based on these results it would appear that 30 minutes was a sufficiently long application of air so that dewatering had reached a steady state, and one would not expect the results to be affected by differences in resistance to flow of water around the cellulosic fibers or fines. As shown, the water retention per unit mass of cellulosic material was approximately 2 grams water per gram of solids, for the fibers and the fines from the same refined kraft pulp. Based on the appearance of Fig. 5, slightly higher water retention might be expected in the case of fines due to their higher external surface area and the likelihood that a film of water remains on those surfaces.

3.2 Dewatering rate affected by kraft pulp fines. As shown in Fig. 6, the secondary fines from refined bleached hardwood kraft pulp had a much larger effect on dewatering rates, compared to the same level of primary fines (from unrefined pulp). The same "default" fibers (from recycled paper) were used in all three of the tests represented in the figure. These results are consistent with those obtained in some previous studies[9,12]. The greater effect of the secondary fines is attributed to their slender shape. It is reasonable to expect that slender, fibrillar fines should be more flexible, have a higher external surface area per unit mass, and have a greater tendency to block channels in the wet paper, when compared to the more blocky shape of primary fines.

Figure 7 shows that the rate of water release slowed dramatically with increasing proportions of secondary fines. In addition, it is apparent that the rate of water release tended to slow down considerably after the first 5 to 10 seconds. The slow-down can be explained, first of all, by the absence of a fiber mat at the very beginning of the

dewatering process. The process of forming a mat capable of significantly slowing the dewatering rate can be understood as having two parts. In the first part of the process, fibers long enough to be filtered by the forming screen form an initial mat. Secondly, it is reasonable to expect that fines become sieved by the mat of fibers, yielding a strong reduction in permeability, which depends on the level of fines in the mixture. As proposed earlier,[8, 9, 12] one way to explain the increased resistance to dewatering with the passage of time is by the ability of cellulosic fines to plug channels within the freshly-formed mat of fibers.

Results not shown, but corresponding to Fig. 6, were also obtained with different levels of primary fines. However, even at the 40% level of primary fines, the effect on dewatering was no greater than that with just 20% of the secondary fines. Thus, the additional test results supported those in Fig. 6 and showed that the differences between the effects of primary and secondary fines were not limited to a specific proportion of fines in the mixture.

The test results reported in Fig. 8 help to show the effect of fiber flexibility. Though, in terms of the fines content, the tests were identical to those reported in Fig. 6, the fiber fraction had been extensively refined. The freeness level of 100 ml CSF at the end of the refining process can be taken as a rough indication of the extent of refining; however, it should be kept in mind that primary fines previously had been removed from the pulp. In addition, most of the secondary fines were removed at the end of the refining process, after the freeness measurement. When comparing results in Figs. 8 and 7 it is important to note the different scales on the vertical axes; the more flexible refined fibers resulted in much slower dewatering, even when no additional secondary fines were added. The slower dewatering in the absence of fines, compared to Fig. 7, can be understood in terms of the ability of the more conformable refined fibers to form a dense mat, leaving narrower passages for the flow of water. It is notable, however, that when the fines content was raised to the 40% level, the dewatering results were about the same for the two sets of tests in Figs. 7 and 8, again indicating a potentially dominating influence of the fines fraction on dewatering rates.

3.3 Retention of kraft fines in the fiber mat. Further insight regarding the mechanism of fines effect on dewatering can be gained by considering the efficiency with which fines are collected in the fiber mat during the dewatering process. As shown in Fig. 9, there was a nonlinear trend in filtrate turbidity *vs*. fines content in the suspension. The non-linearity implies that the fiber fines had a self-retaining tendency. This can be attributed to a denser pad structure and smaller channels within fiber mats that had a relatively high proportion of fiber fines. Such a mechanism would not be expected to be as important at relatively low levels of fines, since one would expect there to be a multiplicity of alternate channels for the water to take as it passes through a fiber mat with a low level of fines. From the appearance of Fig. 9 it would appear that above a level of 30% fines content the fiber mat structure was sufficiently saturated with fines so that the filtration efficiency of the mat became notably higher.

3.4 Cellulosic fines from chemithermomechanical pulp (CTMP). In an effort to test the generality of the conclusions mentioned above, related experiments were carried out with a contrasting class of cellulosic fines obtained from CTMP. Figures 10-

12 show micrographs of three different size classes of these fines. While the figures do show a considerable amount of fibrillar material, much of the fines can be described as "parts of damaged fibers." As one progresses from the R100 (retained on a 100-mesh screen) to a R200, then to the P200 fraction (in the sequence of figures), there is a trend towards smaller particles, but in all cases one sees a wide diversity of shapes.

Figure 13, which is based on laser diffraction analysis, shows relatively wide distributions of particle size. The greatest contrast was between the other samples and P200, which had a modal particle size of about 50 μm, compared to 100-350 μm for the other three fractions that were compared. Modal particle size was taken to be the highest point in the graph of each distribution. The wide distribution of size, as well as the diversity of shape can readily be understood as being a consequence of mechanical fragmentation, as the fibers are torn away from the wood chip and from each other during the refining process. Though thermomechanical pulping has been noted for achieving less reduction in fiber length, compared to stone groundwood pulping, it is clear that many fibers were shortened.

3.5 Dewatering rates affected by CTMP fines. As shown in Fig. 14, resistance to dewatering increased with decreasing modal size of the fines, which were 30% of the dry mass of solids in all cases considered. One of the best-known explanations for this kind of observation has been related to the increasing surface area per unit mass with decreasing size of particles in a filter bed.[6-8]

In order to quantify such a relationship, the smooth lines in Fig. 14 were used to estimate the amount by which different classes of CTMP fines, all at the 30% level, increased the time required to collect 700 ml of filtrate. These results were compared with the modal particle sizes obtained from Fig. 13. As shown in Fig. 15, the results fell approximately on a straight line passing through the origin. Thus, to a first approximation, resistance to dewatering increased in inverse proportion with the particle size, as estimated from the modal diameters determined by laser diffraction tests. These results can be explained in terms of a mechanism in which small, unattached fines are able to migrate through the fiber mat and get stuck in positions where they have relatively large effects on mat permeability. Smaller particles would be expected to have a larger impact, according to such a mechanism, due to their higher surface area per unit mass and due to their greater ability to change their locations relative to the adjacent fibers in the mat.

4. Conclusions

Cellulosic fines collected from unrefined bleached hardwood kraft, from refining the fiber component of the same kraft pulp, and also from chemithermomechanical pulp differed considerably, based on optical microscopy and also based on laser diffraction particle size analysis. Resistance to gravity dewatering generally increased with decreasing size of the fines. In addition, the highly fibrillar secondary fines from refined bleached hardwood kraft fibers caused a much greater reduction in dewatering rates than the more compact primary fines obtained from the corresponding unrefined hardwood. Results generally support a hypothesis in which fines become entrapped in the mat of fibers that is collected during a dewatering experiment. Once trapped in the fiber mat,

the fines contribute to drainage resistance in inverse proportion to their size, as determined by the laser diffraction method.

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Figures

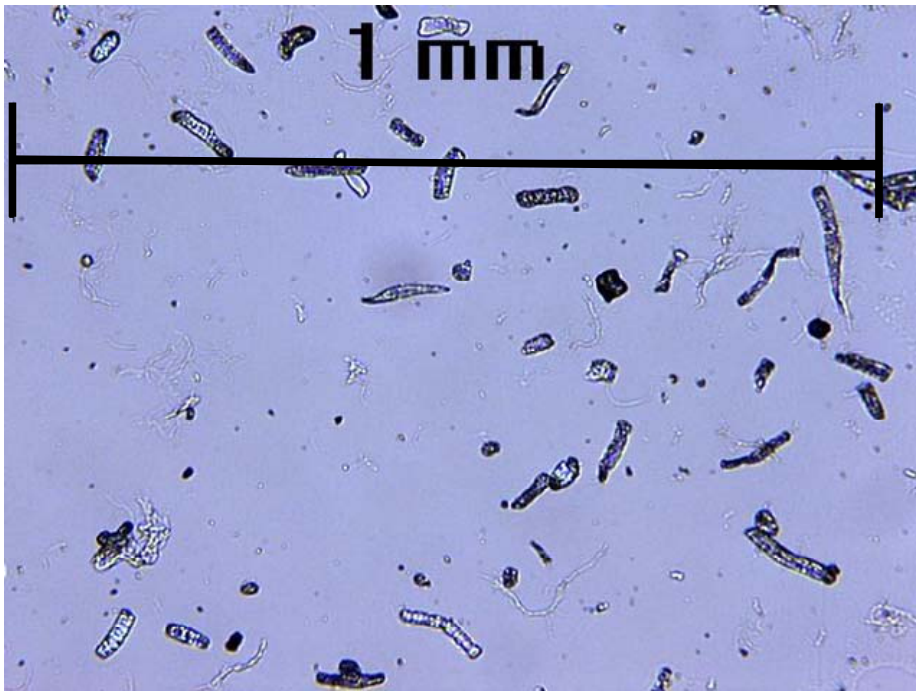

Figure 1. Micrograph of primary fines (collected before refining) from bleached hardwood kraft pulp.

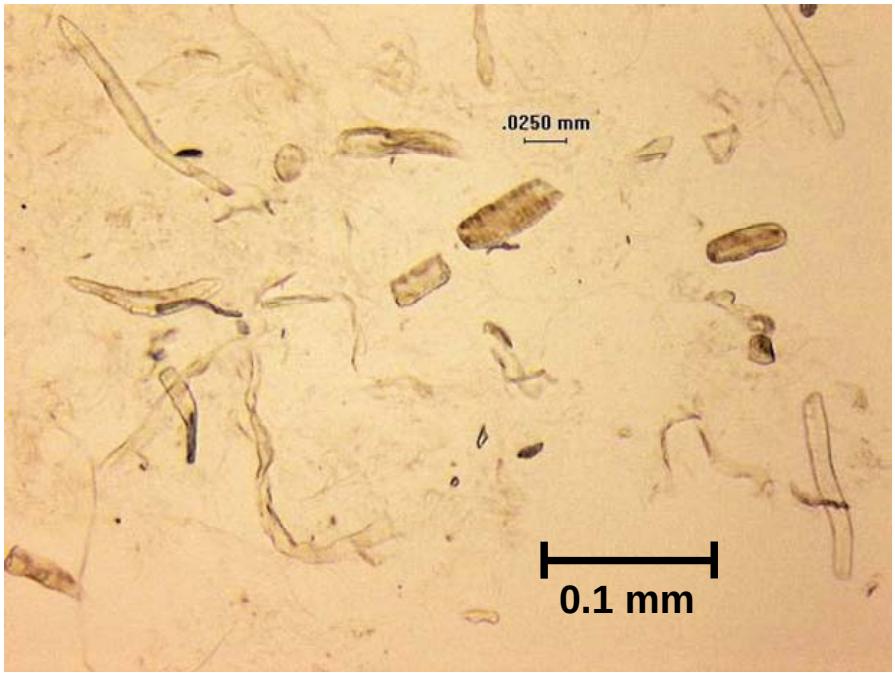

Figure 2. Micrograph of secondary fines (collected after refining fines-free pulp) from bleached hardwood kraft pulp refined to 100 ml Canadian Standard Freeness.

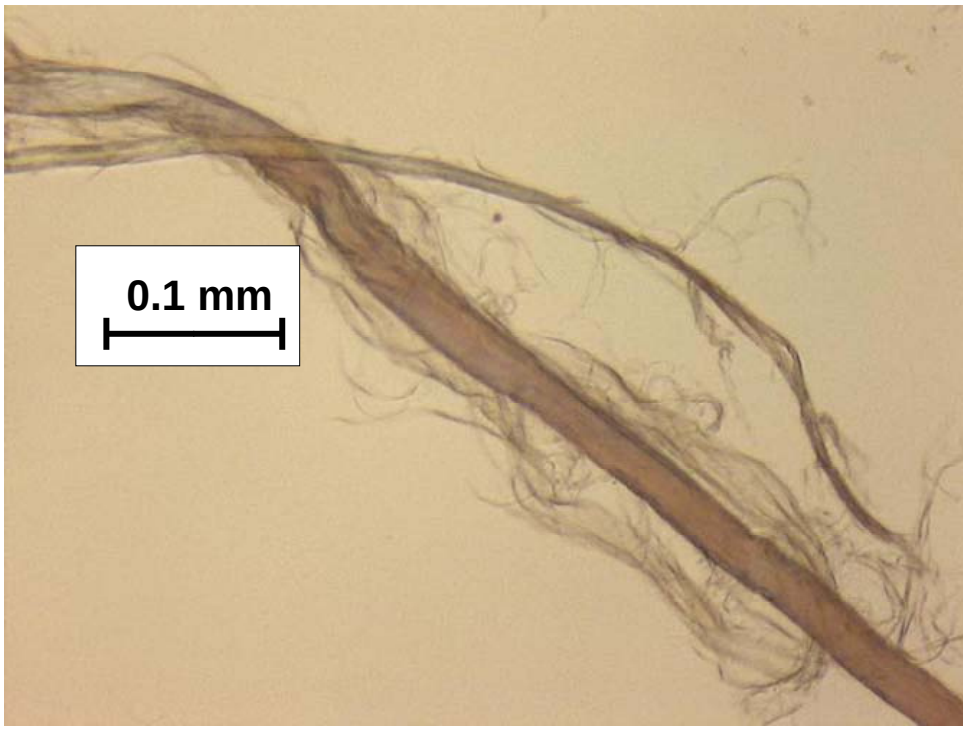

Figure 3. Micrograph of highly refined kraft fibers from the batch that was used in preparation of the secondary fines. Note the fibrillation at the fiber surfaces.

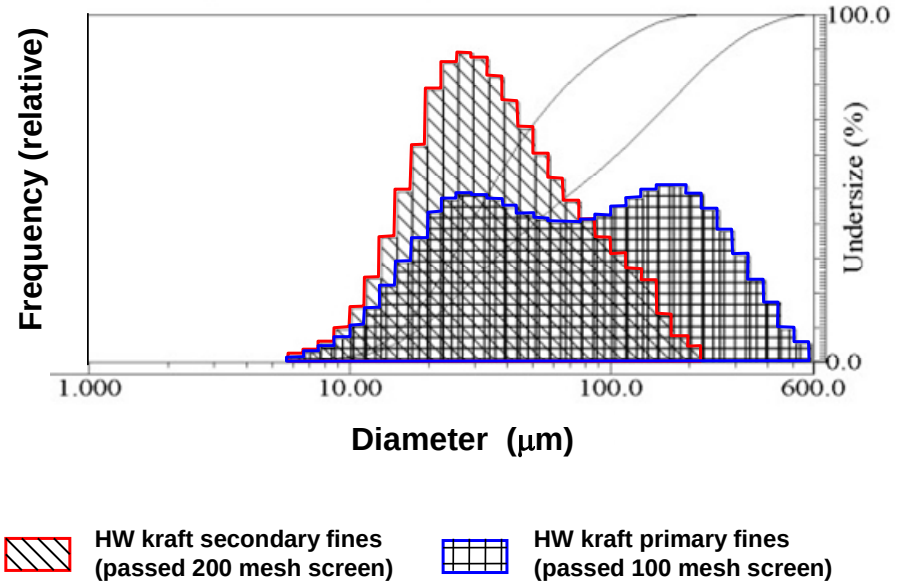

Figure 4. Apparent particle size distributions of primary and secondary fines from bleached hardwood kraft pulp, based on laser diffraction analysis.

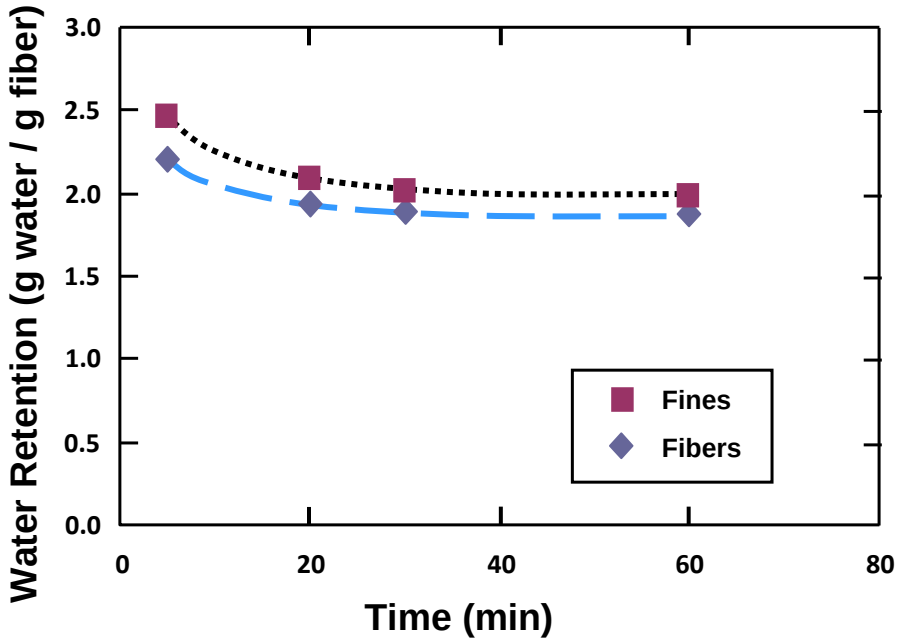

Figure 5. Results of modified water retention tests, using 68.9 kPa air pressure to remove water from between fibers or fines in a pad, followed by weighing, oven-drying, and reweighing.

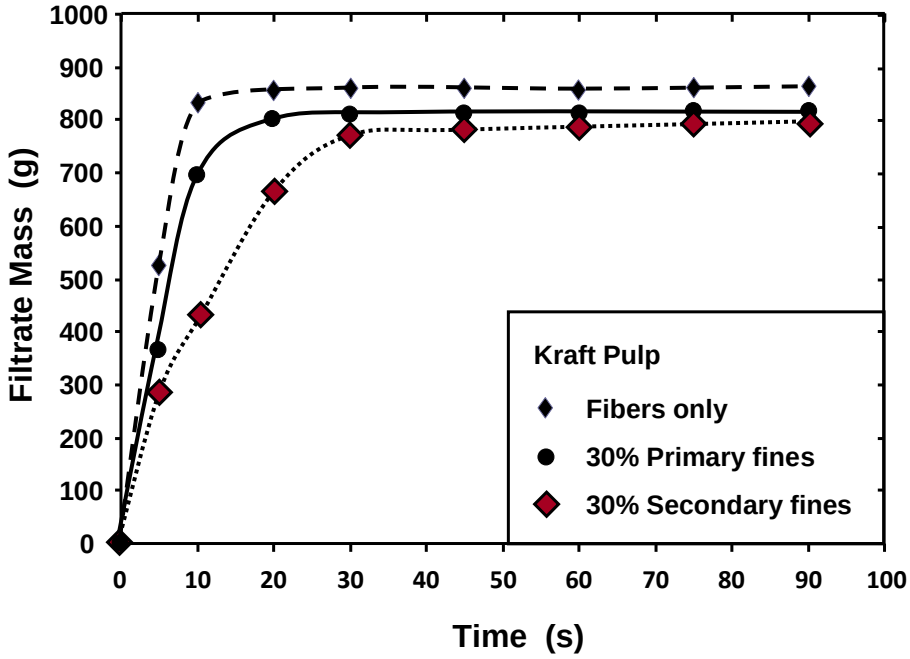

Figure 6. Gravity filtrate volume vs. time for 0.5% solids suspensions (1000 ml each) composed of 30% fines (passing a screen either before or after refining) with 70% by mass of a default sample of recycled kraft fibers.

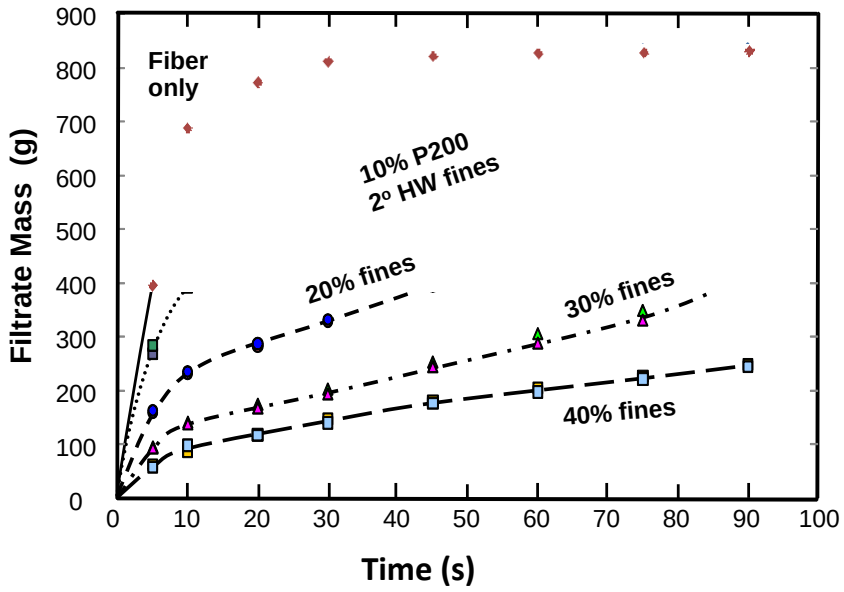

Figure 7. Gravity filtrate volume vs. time for 0.5% solids suspensions (1000 ml each) composed of different proportions of secondary fines with a default sample of recycled kraft fibers.

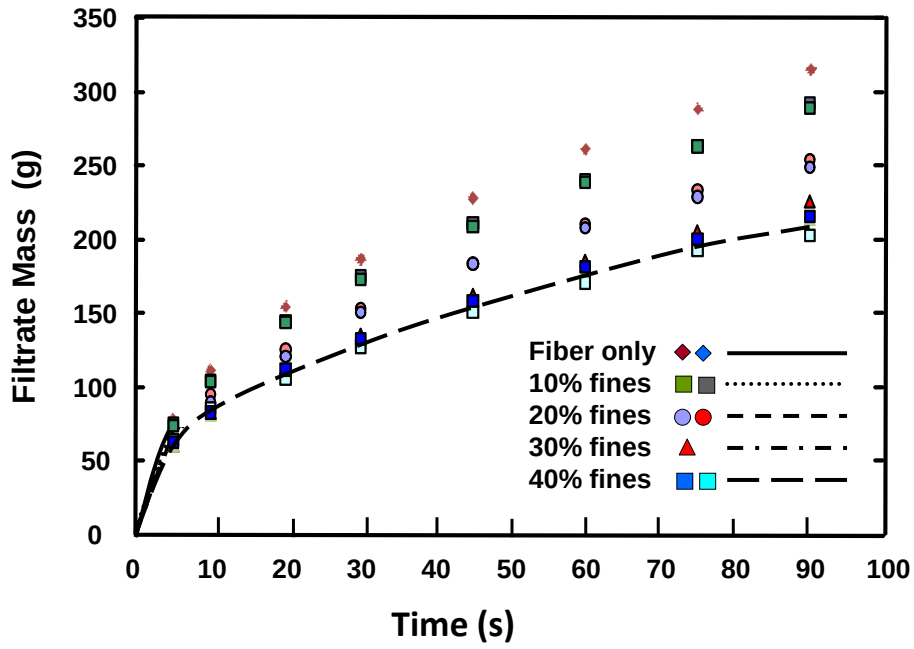

Figure 8. Gravity filtrate volume vs. time for 0.5% solids suspensions (1000 ml each) composed of different proportions of secondary fines with highly refined kraft fibers.

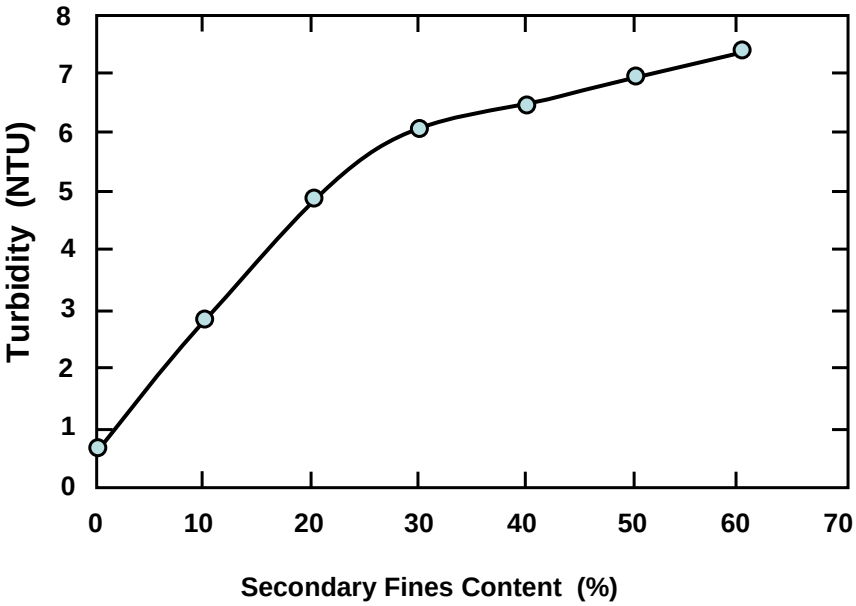

Figure 9. Effect of bleached kraft secondary fines content in the fiber slurry on the turbidity of filtrate collected after freeness tests.

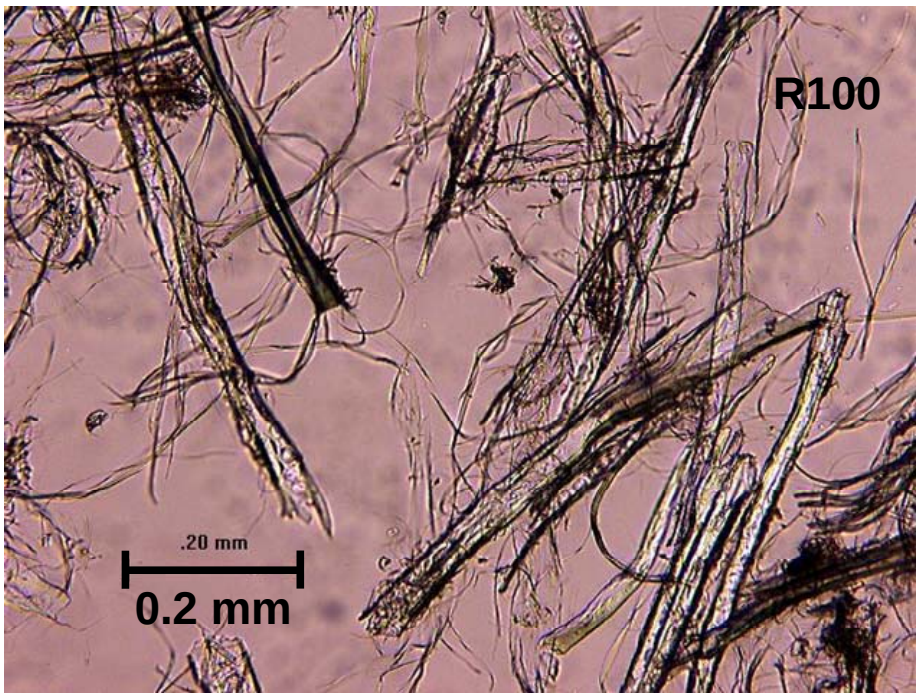

Figure 10. Micrograph of a coarse fraction (retained on a 100 mesh screen) from Bauer-McNett classification of chemithermomechanical pulp (CTMP).

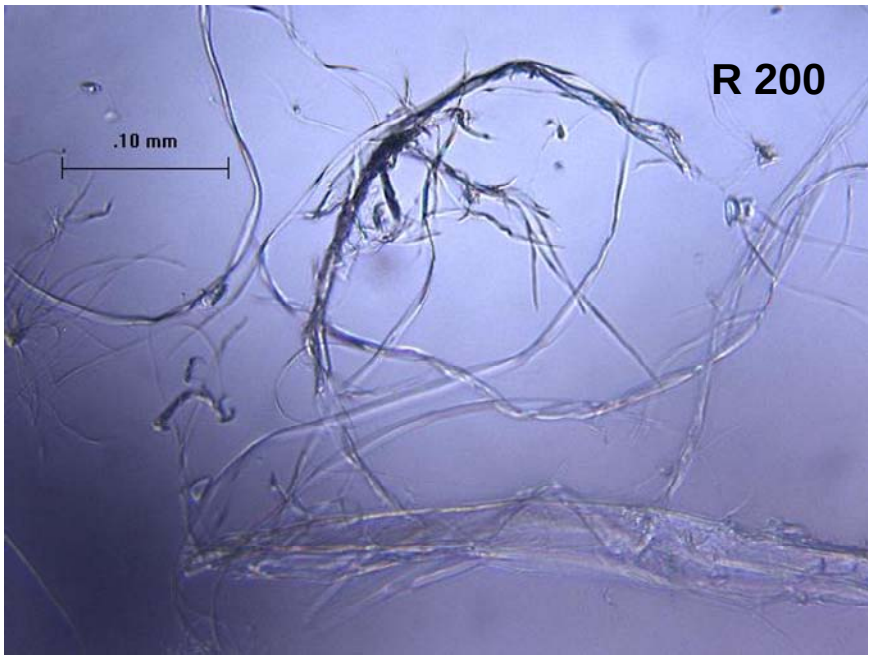

Figure 11. Micrograph of an intermediate fraction (retained on a 200 mesh screen) from Bauer-McNett classification of chemithermomechanical pulp (CTMP).

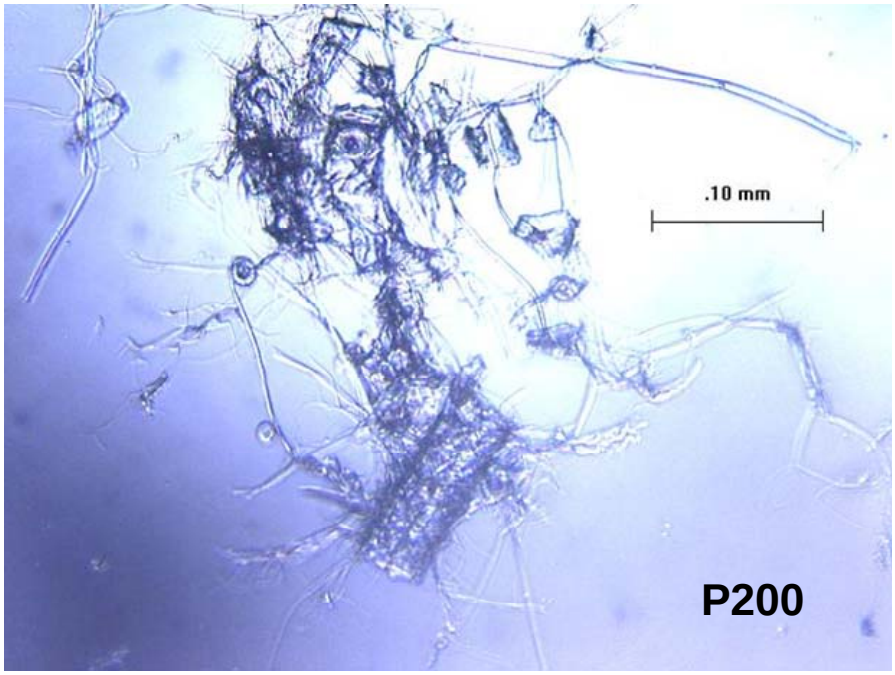

Figure 12. Micrograph of a fine fraction (passing a 200 mesh screen) from Bauer-McNett classification of chemithermomechanical pulp (CTMP).

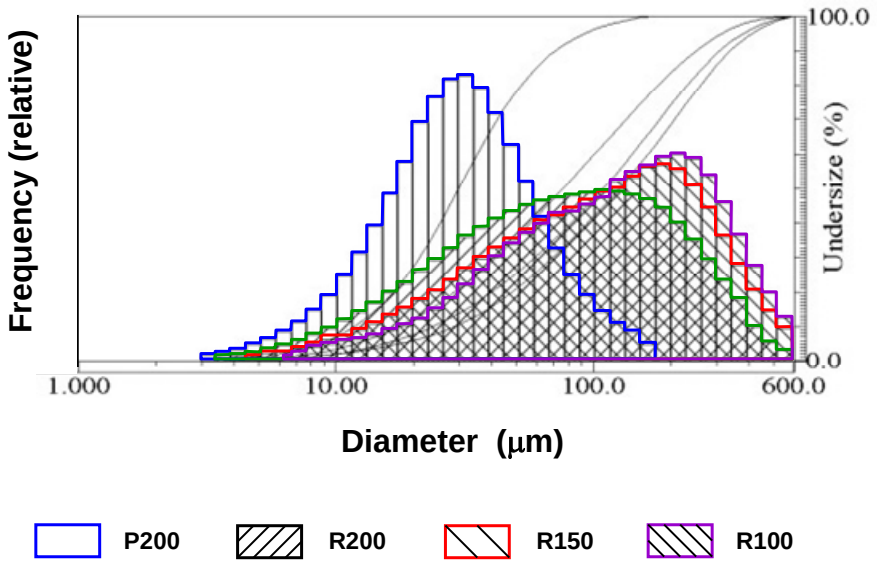

Figure 13. Apparent particle size distributions of chemithermomechanical pulp (CTMP) fractions, based on laser diffraction analysis.

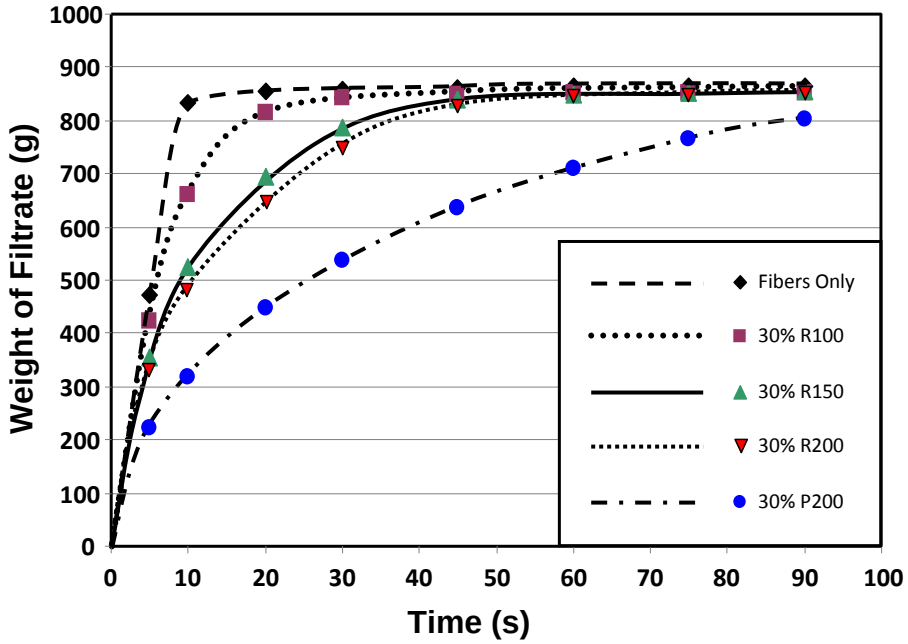

Figure 14. Gravity filtrate volume vs. time for 0.5% solids suspensions (1000 ml each) composed of different size classes of fines from chemithermomechanical pulp (CTMP).

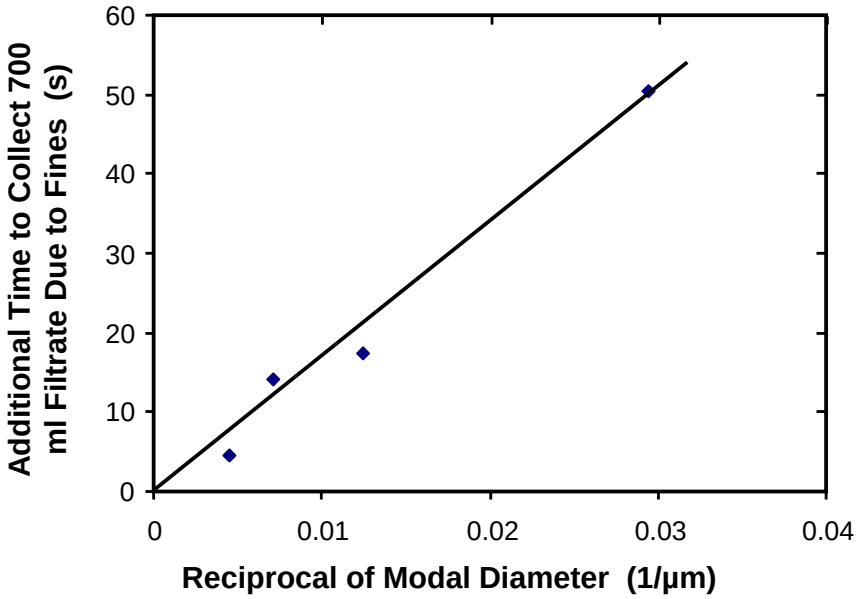

Figure 15. Plot of the effect of fines on the dewatering time to collect 700 ml of filtrate, compared with the reciprocal of modal particle size, based on laser diffraction tests. Note that the times were estimated from Fig. 7 by subtracting "7.5 seconds," the value corresponding to fibers alone.