Focused beam reflectant measurement as a tool to measure flocculation

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ABSTRACT: This paper presents several uses of *focused beam reflectance measurement* (FBRM) as tool to measure flocculation. It includes information on the interaction of wet-end aids with the different pulp fractions, such as optimal polymer dosage and the resulting floc properties (floc size, strength, etc.). The study of the floc evolution over time under different conditions also allows us to identify flocculation mechanisms. The FBRM methodology provided a real-time, in-process monitor to establish the evolution of the resulting flocculation state and polymer efficiency under the influence of shear forces. The evaluation included a study of deflocculation and reflocculation processes occurring in the sample. We also looked at the problem of contaminants accumulation, as can occur with increased use of recovered paper and the closure of water systems. In addition, FBRM has been used to identify dissolved and colloidal material agglomeration and to study its influence on polymer demand for a given furnish.

Application: The results from this study help explain flocculation processes and can help obtain real-time data as a first step in controlling the flocculation process in a mill.

Flocculation control is becoming increasingly important in the industry. Papermakers want "optimum flocculation," with good retention, good drainage, and good formation [1-3].

Flocculation optimization on a machine often begins with the monitoring of consistency, retention, and charge. While there are several on-line wet-end chemistry instrumentations available for consistency, retention, and charge monitoring, the area of flocculation is still under development [4-5]. Thus, a flocculation sensor to directly control flocculation would considerably improve the control process. At the moment, this has to be indirectly controlled through the retention, consistency, and charge values. Charge also indirectly controls dissolved and colloidal material concentrations.

Traditionally, papermakers optimize the dosage of polymers with techniques based on the electronic charge of the particles. The principles and limitations of available instruments have been recently published by Hubbe [6]. Flocculation phenomenon results from an increase in the size of the particle aggregates and the inherent consequences and behavior of these agglomerated constituents. These effects are a function of each individual flocculant type. Therefore an approach to indirectly monitor flocculation is to focus on the common parameter for all flocculation mechanisms: the change in both the average size and the size distribution of the suspended particles, aggregates, or flocs present in the pulp suspension. This procedure has been already used to study chemical flocculation [7-9], the breakdown of flocs under the influence of hydrodynamic shear [10-11] and it has been also proposed by Nalco as an on-line sensor to monitor paper machine retention [12-13]

This paper presents monitoring of the particle dimensions and populations of various pulp suspensions as well as the fractions of the furnish under different conditions in order to understand the interactions between various polymeric additives and the different pulp fractions. The particles/polymer interactions are investigated as a function of the process shear forces, time, and flocculant dosage. The methodology is based on a technique called non-imaging reflectance scanning laser microscopy (SLM) or focused beam reflectance measurement (FBRM).

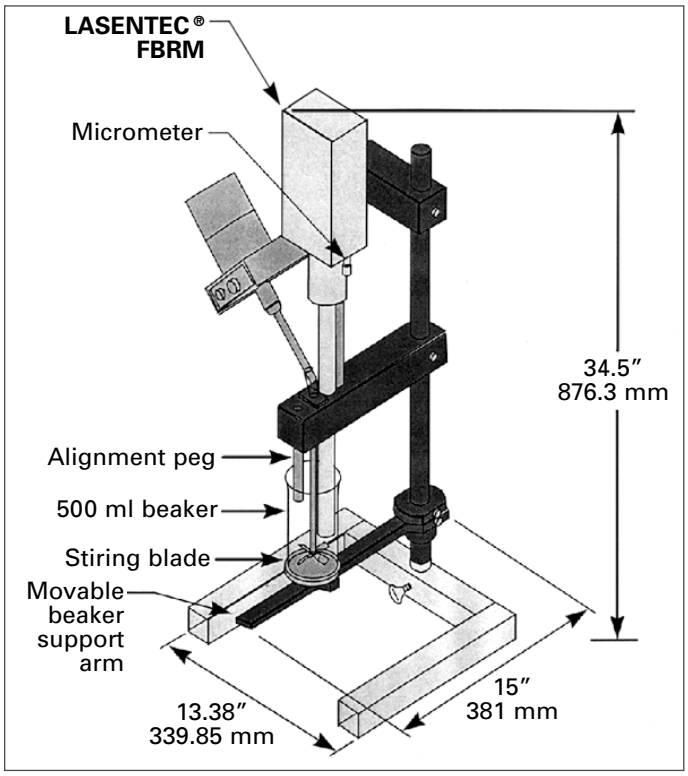
EXPERIMENTAL PROCEDURE

For measurements in this study, we used a commercially available non-imaging scanning laser microscope or focused beam reflectance measurement (FBRM) system (LASENTEC FBRM M500LF), which is shown in **Fig. 1**.

The FBRM instruments operate by scanning a highly focused laser beam at a fixed speed across particles in suspension and measuring the time duration of the backscattered light from these particles. To acquire the best data it is necessary to optimize the following parameters: probe location, duration of the measure, and data averaging [14-15].

With the FBRM software, we can obtain many different data as graphical representations of chord length distribution in a certain time, graphical representations of the evolution of average parameters and statistics with time, spreadsheets with all the parameters and statistics over time, and so forth. We can also compare results with previous experiments and introduce additional data from other external sources (thermometer, pH meter). With these data, we were able to study the particle size distribution and evolution of flocculation and agglomeration processes. We could determine the minimum quantity of flocculant needed to begin the agglomeration of a given pulp fraction and the operative flocculation mechanisms and their evolution. We also could study the flocs resistance to shear, the behavior of the different types of flocs under the influence of shear forces, reflocculation, and other issues.

As mentioned, papermakers are dealing with an increase in detrimental substances in process systems. These substances are mainly dissolved and colloidal materials that may be potential contaminants, although when they are in a dissolved or colloidal state they move around the system without being especially problematic. The problems start when they are destabilized, forming aggregates due to a shock of pH, conductivity, temperature, or through interaction with polymers already present in the system. The methodology developed for this study allow us to observe the destabiliza-



1. Schematic representation of the M500LF FBRM system.

tion of dissolved and colloidal material (DCM), opening new possibilities to understand destabilization conditions and to optimize control during papermaking.

For our experiments, we set the instrument data acquisition time at 10 seconds per data point and fixed the probe in the vessel. We did not average or smooth the data. We connected the instrument to a commercially available compact titrator in order to add the volume of flocculant in regular time intervals. A computer controls both instruments.

This study focused on three examples of FBRM application as a tool to study flocculation—white water flocculation, flocculation mechanisms and flocs properties, and DCM destabilization.

RESULTS

Flocculation of white waters

With the aim of determining the usefulness of FBRM to study flocculation processes in white waters, we carried out a series of experiments using two white waters with different concentrations of DCM.

We chose unbleached softwood as the fiber source and used the two disintegrating conditions shown in **Table I** to obtain the white water. We used disintegration additives to obtain a higher DCM in the white water and to study its effect on polymer demand. After disintegration, we filtered the stock through a 150-mesh wire using a dynamic drainage jar (DDJ) in order to acquire the white waters.

For both experiments, we inserted the FBRM probe into a 500 mL beaker containing 100 mL of sample. We stirred the sample at 250 rpm during the experiment, while gradually adding flocculant with an automatic dosing unit that had a predetermined addition sequence. In this series of experiments, we chose a commercial cationic PAM as the flocculant to use. After

	WITH ADDITIVES	WITHOUT ADDITIVES
Consistency (%)	3.5	3.5
Volume (L)	2	2
T (°C)	45	45
Speed (rpm)	3000	3000
Additives	1% NaOH, 1% Soap, 1% H_2O_2 2.5% Silicate	1% NaOH
Time (min)	20	20

I. Disintegration conditions of the pulp.

2 min of agitation at 250 rpm, we added the polymer at a rate of 0.016 meq every 30 seconds during the first 5 min and then at a rate of 0.16 meq every 30 seconds.

Figure 2 shows the results, including the number of chords per second less than 30 µm and the number of chords between 200 and 500 µm versus the flocculant dose in meq/L. We chose the two intervals as the most representative of the fraction fines-fillers-DCM (the lower one) and the resulting flocs (the higher one). In both cases, the number of chords less than 30 µm decreased while the number of chords between 200-500 µm increased. This revealed the flocculation process. The fines, fillers, and DCM (whose chords are lower than 30 µm) were agglomerated due to the formation of bridges by the polyacrylamide. This agglomeration event results in larger flocs and is typical behavior of this polymer. In our case, this technique detects these flocs as large size chords (200-500 µm). After a certain polymer dose, the curve of the chords lower than 30 µm stabilized and the large chords stopped increasing. This phenomenon indicated the end of the flocculation of small particles. Although not shown in this report, the higher dosage of PAM resulted in further aggregation of flocs. The flocs were larger than 500 µm while simultaneously the number of particles between 200-500 µm decreased.

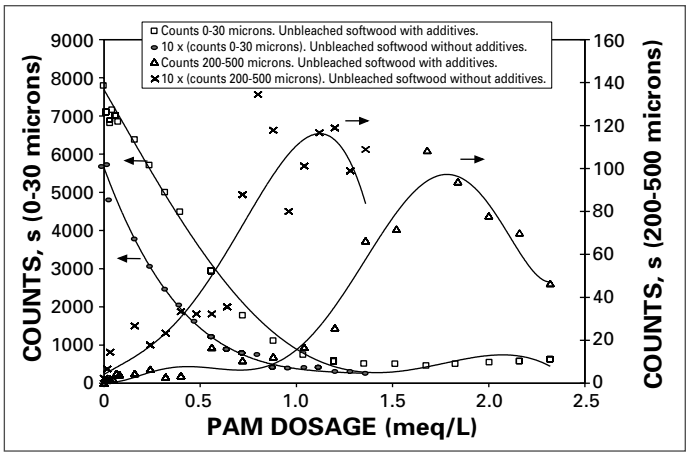
When comparing the two curves for small and large chords, we note that white waters obtained with disintegration additives consumed significantly more flocculant. This clearly indicates that there was more dissolved and colloidal material following incorporation of disintegration additives, as expected.

This simple but clear example shows the possibility of using this technique for studying flocculation processes of pulp suspensions, and allows us to obtain information about the different fractions present in the mixture. It also reveals the influence of DCM on polymer consumption (discussed later) and allows us to study issues such as the effect of water closure on retention aids efficiency and to look for polymers that are not affected by DCM accumulation.

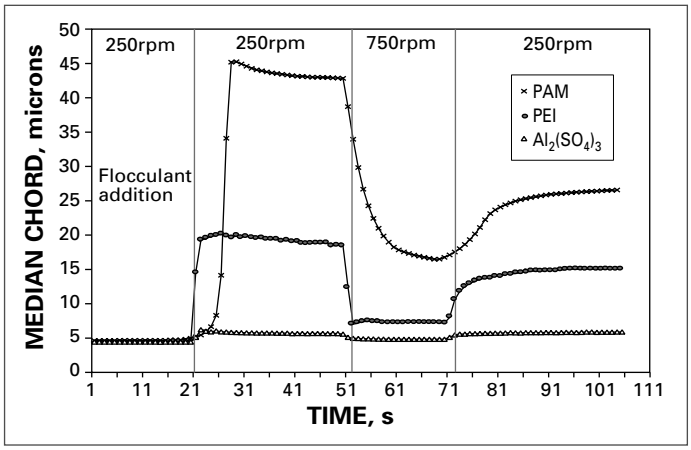
FBRM study of flocculation mechanisms and flocculation properties

With the aim of determining the possibility of using FBRM to study flocculation mechanisms, we chose three distinctly different flocculants for our experiments. They included a cationic polyacrylamide (PAM), polyethyleneimine (PEI), and aluminum sulfate. The PAM was a low charge density and high molecular weight that flocculates by formation of bridges. The PEI was a high-charge polymer that acts by a patch mechanism. The aluminium sulfate flocculates by electrostatic neutralization.

We included calcium carbonate—a frequently used filler in paper manufacturing—as part of the mixtures studied. The selected retention additives are absorbed on the mineral fillers in different ways, depending on the nature of the flocculant, and



2. Disintegration condition influence on flocculation of white waters.



3. Evolution of the flocculation process with different flocculants.

therefore produce three types of flocs, depending on the predominant flocculation mechanisms.

To simplify the study, we used a 1% calcium carbonate suspension (10g/L) and did not add contaminants. The flocculant dosage for PEI and sulfate was the theoretical volume obtained with a charge detector PCD03. For PAM, we used FBRM measures, since neutralization is not the main flocculation mechanism [*10*].

The experiments for the three flocculants followed the same procedure. We placed the FBRM probe into the suspension that was then stirred for 2 min at 250 rpm. During this time, the measurement of the FBRM was constant, and indicated the dimension and population of the mineral particles in suspension. After the 2 min of stirring, we added the flocculant. After 5 min of observation, we increased the stirring speed to 750 rpm for 2 min to induce deflocculation, with the aim of studying the behavior of the flocs formed in relation to the shear forces. Then, to determine the evolution of the reflocculation for the different flocculants, we reduced the speed to 250 rpm for several more minutes.

Figure 3 shows the evolution of the median chord size versus time for the three different flocculants. As expected, the dimensions of the particles increase due to the flocculant effect and we obtained different floc sizes for the three additives investigated. When shear forces were increased, the flocs were broken as evidenced by the reduction in the median size. Finally, reflocculation is achieved when shear forces decrease again to the initial levels. The reflocculation level reached for each polymer is different. For aluminum sulfate, the median chord returns to the pre-high shear value expected due to the neutralization mechanism of flocculation. For PAM and PEI only partial reflocculation is achieved. The deflocculation curves and the reflocculation curves yield information about the shear resistance of the different flocs and the reversibility of the flocculation process, and therefore about the predominant flocculation mechanism.

vThe size of the flocs differs in each case, due to the diverse flocculation degree produced. PAM produced larger flocs because of the nature of the polymer and its flocculation mechanism (formation of bridges). PEI produced intermediate size flocs, aluminum sulfate produced the smallest flocs.

The behavior of the different flocs also differed. When using aluminum sulfate, the flocs are easily destroyed as stirring speed increases, almost returning to the same grade of flocculation when the stirring conditions are over. However, when using PAM, the flocculation is slower. Furthermore, the maximum size is reached at longer times than for PEI or aluminum sulfate. This is likely the result of this being the bridge mechanism of flocculation. Specifically, at first the polymer is absorbed on the filler particles forming tails and loops and as a second step these tails and loops will interact with other particles forming the aggregates. When we increase the shear forces, the tails and loops of the polymer are broken and therefore when the shear forces decrease again reflocculation takes place only partially since the polymer has been affected. PEI shows an intermediate behavior, due to its mechanism of patching formation. Due to the polymeric nature of the PEI, shear forces can partially affect the polymer so that the reflocculation level is also lower than the original flocculation degree.

Alfano and co-workers have studied floc breakage at a constant shear rate by adjusting the data from the FBRM to an exponential equation (11). In a similar way the deflocculation curves corresponding to the flocs splitting when the shear rate was increased have been adjusted to the following exponential equation:

$$y = Co + A^* EXP(-t / Tb) \quad (1)$$

Furthermore, in this work, the reflocculation curves in which the median cord size increases once the shear forces that produce the break down of the flocs have decreased, were adjusted to the following equation:

$$y = C\infty - K * EXP(-t / Ts) \quad (2)$$

Where "y" is the median chord size (micrometers), "t" is time in seconds and Co, C∞, A, K, Tb and Ts are the parameters of the curve.

As a matter of practicality in data analysis, the following exponentials described the curves:

$$Y = A * EXP (- B * t) \quad (3)$$

for the deflocculation process, and:

$$Y = K * EXP (- S * t) \quad (4)$$

for the reflocculation process.

When comparing equations 1-3 and 2-4, we obtain B= 1/Tb and S= 1/ Ts.

The values of Co and C∞ were determined with interactions, until finding the value that gave a better adjustment of the deflocculation curve to the experimental

points after subtracting that value. To adjust the reflocculation curves of the median size, the same scheme was followed, although the subtraction has to be done the other way round, taking the value of C∞ and subtracting the value of the original curve.

This method did not reliably reproduce the beginning of the curve (where the variation of the median size is bigger), mainly because it was affected by the change in stirring, as this was not instantaneous and took about 10 seconds. That is why the first two points of the curve have a higher experimental error.

Tables II and III show the parameters obtained as well as the correlation coefficients of the adjustments.

During flocculation there is a dynamic equilibrium between the formation and the breakage of flocs. In our case, the value of Tb obtained is the global result of these two phenomena. Tb is a way to measure simultaneously the difference between the rate of formation and breakage of flocs. A high value of Tb can be due to a high equilibrium grade (although the flocs are very soft) or to a high strength of the flocs (even if this means a reflocculation rate almost null). In this way, we could affirm that Tb is a measure of the global resistance of the flocs, considering this as a combination between the strength of the flocs itself and the dynamic reflocculation rates that take place without changing the conditions.

A large decrease in the curves occurs when shearing forces are increased. This is due to the high rate of floc splitting. Flocs initially formed under gentle agitation that minimized floc breakage. Moreover, the concentration of colloids was at a minimum because of the high extent of flocculation. This is why in the curve we mainly see the splitting rate. The reformation rate is almost null because practically no flocs are broken yet. At this point Tb values are mainly influence by the initial strength of the flocs. Therefore at higher strength, higher Tb if dynamic reflocculation is negligible, as for example in the bridge flocculation mechanisms produced by the PAM. However, the value of Tb is slightly higher with aluminum sulfate than when using PEI. In this case, dynamic reflocculation is not negligible, the superior reversibility of the flocs formed partially compensates the effect pointed out before. In this way, as soon as some broken flocs are produced, the rate of formation of new flocs (originated from the broken ones) starts to increase, enlarging the final value of Tb. This occurs even though the concentration of colloids is almost zero at the beginning of the curve. As aluminum sulfate produces a neutralization mechanism with a total reflocculation degree, the Tb value is higher than for PEI.

As reflocculation takes place at 250 rpm, Ts can be used as a way to measure the reflocculation speed after the influence of high shear forces. The lower values of Ts represent the more efficient and faster reflocculation.

The reflocculation is faster with aluminum sulfate, as confirmed by the value of Ts being lower. This means that these are the most "reversible" flocs. The intermediate value corresponds to PEI and the higher one to PAM, indicating the flocs are less reversible as corresponds to a mechanism of bridges. This information could be complemented by comparing the size of the final flocs after reflocculation and the maximum size obtained initially. In the case of PAM, only a 60% of the maximum size is regained, while 80% of the maximum size is approximately recovered with PEI and almost 100% recovery is obtained when using aluminum sulfate. These data show the interest of this procedure to study flocculation mechanisms.

FLOCCULANT	Co (µm)	A (µm)	Tb (s)	r^2
$Al_2(SO_4)_3$	4.82	0.48	27.9	0.972
PEI	7.21	8.32	19.1	0.835
PAM	16.2	28.02	39.8	0.998

Median Chord = Co + A * EXP (- t /Tb)

II. Deflocculation curve adjustments for different flocculants.

FLOCCULANT	C∞ (µm)	K (µm)	Ts (s)	r^2
$Al_2(SO_4)_3$	5.85	0.72	68.97	0.89
PEI	15.15	6.26	69.44	0.93
PAM	26.6	16.66	75.76	0.99

Median Chord = Co - K * EXP (- t /Ts)

III. Reflocculation curve adjustments for different flocculants.

With the aim of achieving a deeper knowledge about the behavior of the flocs, we conducted another series of experiments to study their resistance and their reversibility. In this case, the methodology was the same as that used to generate the results of Fig. 3. Thus, we studied the evolution of flocs obtained with PAM at 250 rpm, but the break down of flocs was carried out at different stirring rates. Five different speeds were chosen: 500, 750, 1000, 1250, and 1500 rpm. **Figure 4** presents the FBRM measures obtained. We can observe they are hard flocs, as they need high shear forces to induce significant breakage. Once broken there is some flocculation but never achieving the original state. Once the majority of the flocs are broken, an increase of shear forces does not affect them as suggested by the similar results obtained with 1250 and 1500 rpm.

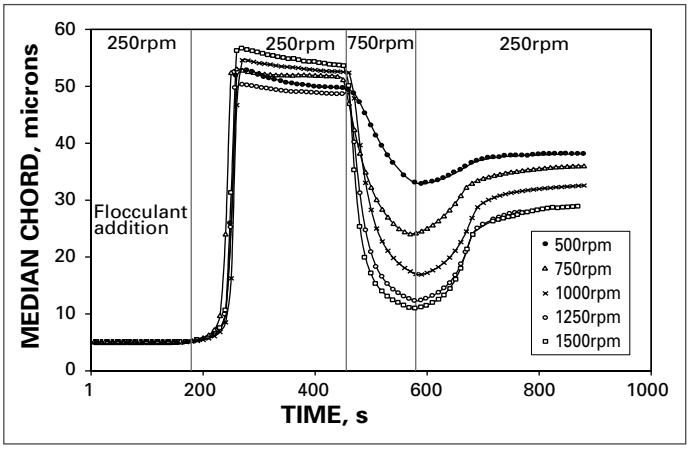
Tables IV and V show the adjustment of the curves (de-flocculation and reflocculation) and their Tb and Ts values. It is clear that when stirring rate increases, Tb decreases as a consequence of the smaller resistance of the flocs at high speeds. Ts also declines when augmenting the stirring rate; the reflocculation rate is higher as the number of broken flocs increased. Reversibility decreases as the stirring speed increases until 1250 rpm, as seen in studying the final size of the flocs, which constantly declines. At this agitation level the flocs have been totally broken down and a further agitation will produce the same effect.

These data are important with respect to predicting the performance and efficiency of retention aids for the new paper machines that operate at high turbulence.

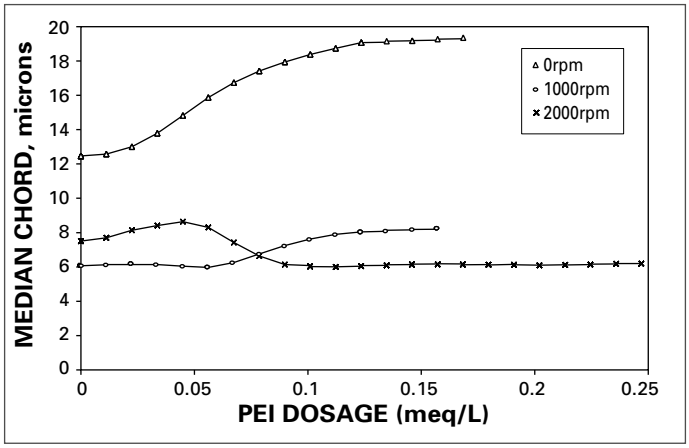
DCM DESTABILIZATION

An important, and also complex, aspect of papermaking is the way in which the interactions between flocculants and DCM (as well as fines and fillers) take place in paper suspensions, and how DCM concentration influences this phenomenon. In order to achieve a deeper knowledge about this issue, we investigated the interaction of polymers and the fines, fillers and DCM obtained from recovered paper.

The fiber raw material used for this study was a mixture of recovered paper that contained different kinds of paper. The white waters were obtained as in example 1. We centrifuged these white waters for 10 min at 1000 and 2000 rpm in order to separate the DCM fraction from fines and fillers. The polymer used for flocculation was PEI.



4. PAM flocs shear resistance.



5. Mean chord evolution for samples centrifuged at 0, 1000 and 2000 rpm.

SHEAR (rpm)	Co (µm)	A (µm)	Tb (s)	r^2
500	28.8	21.238	69.44	0.994
750	22.5	25.088	40.98	0.990
1000	16.0	31.131	33.11	0.999
1250	11.7	37.144	29.67	0.998
1500	10.6	32.491	27.10	0.994

Median Chord = Co + A * EXP (- t /Tb)

IV. Deflocculation curves adjustment for different stirring speeds.

SHEAR (rpm)	C∞ (µm)	K (µm)	Ts (s)	r^2
500	38.1	10.147	49.3	0.991
750	35.5	30.303	45.3	0.991
1000	32.3	38.864	44.1	0.990
1250	28.3	51.883	41.2	0.987
1500	28.2	57.698	40.2	0.987

Median Chord = Co - K * EXP (- t /Ts)

V. Reflocculation curves adjustment for different stirring speeds.

Table VI shows the cationic demand obtained for the three samples. This parameter represents the amount of polyelectrolyte necessary to neutralize the anionic charges of a given sample. Only when the flocculation takes place by neutralization does the value of the cationic demand correspond to the optimal polymer dosage to produce the flocculation phenomena. Therefore, FBRM data are more precise than cationic demand measurements and it gives complementary information.

These data suggest that white water consumed a higher quantity of flocculant, while there was no difference between the results for the two centrifugation rates. This could mean that the fines and fillers fraction was totally removed even at low centrifugation rate, and thus no difference is observed when centrifugation takes place at 1000 or 2000 rpm. However, the studies carried out with FBRM will demonstrate that this is not what happens in reality.

Mean chords evolutions versus polymer dosage were analyzed for all the cases as shown in **Fig. 5**. In this case, we stirred the 100 mL sample at 250 rpm during the experiment. After 2 min of agitation the experiment started and 0.001123 meq. of PEI was added every 30 seconds. The flocculation process has a different evolution for each one of the three samples. With the white water, an increase of the mean chord resulted. For the DCM fraction obtained at 1000 rpm, the mean evolution followed the same performance as the white water. Namely, an increase of the means as the flocculant was added until it achieved a certain constant value. However, after a small increase, the evolution of the mean chord for the sample centrifuged at 2000 rpm was the opposite, decreasing until it reached a constant value.

To justify this behavior, it is necessary to consider other statistical parameters that will allow us to interpret this data. Therefore, we looked at the evolution of different particle size intervals (1-6; 7-25 and 25-100 µm) and the distribution of the chord size before adding any flocculant and at the end of the flocculation process. These values are shown in **Fig. 6**. In the case of the sample that was not centrifuged (fines, fillers and DCM fraction), the flocculation process implied a decrease in the number of chords between 1 and 6 µm and an increase in the number of bigger chords (see Fig. 6A). This result was expected, as the small particles joined together forming aggregates that had larger chords sizes, increasing the value of the mean chord size.

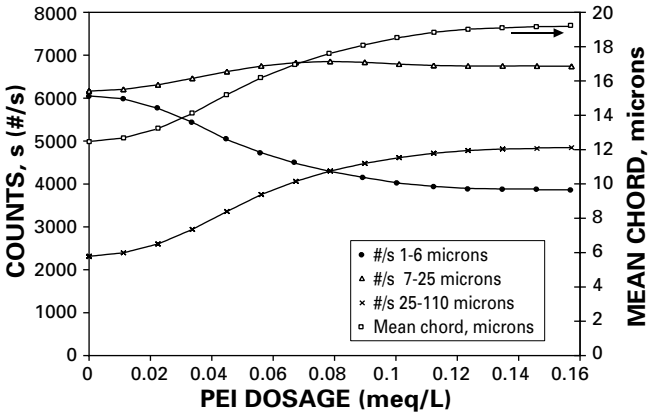
CENTRIFUGATION SPEED (rpm)	CATIONIC DEMAND (meq/L)
0	0.70
1000	0.63
2000	0.63

VI. Cationic demand for different centrifugation speeds.

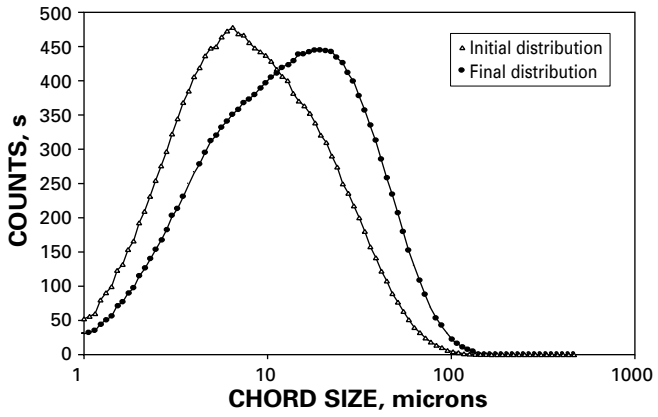
Figure 6B shows that there was a displacement of the chords distribution towards larger sizes.

In the case of the sample centrifuged at 1000 rpm, an increase in the number of counts for all the chord sizes was observed (see Fig. 6C). This means that the number of counts of all three sizes evaluated increased. We observed an greater number of counts (for all the chord sizes) when comparing the first and final distributions (see Fig. 6D). This is due to the impossibility of measuring chords smaller than 0.8 µm by FBRM, causing the non-detection of those dissolved and colloidal particles until they are flocculated producing bigger particles capable of being detected by the technique.

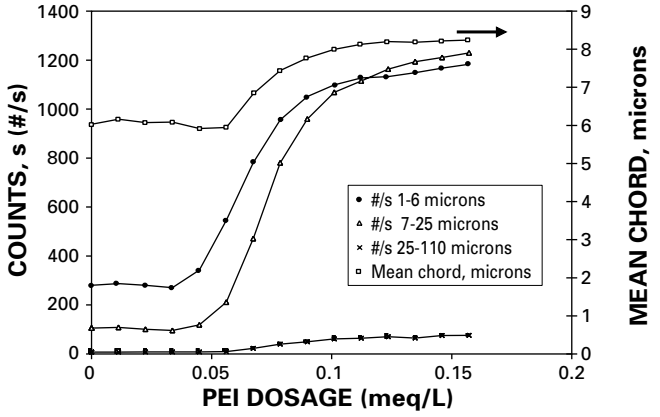
When these materials flocculate, the aggregates formed have chords between 1 and 6 µm, producing an increase in



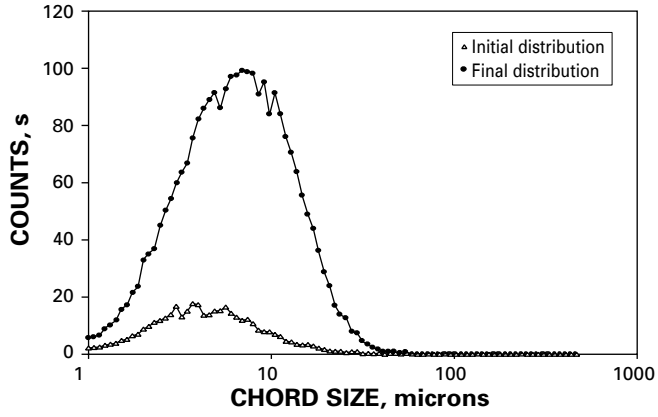
A: Statistics of 0 rpm sample



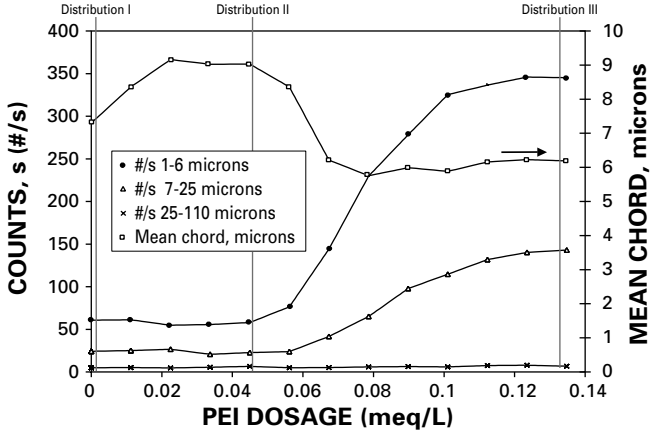
B: Distribution of 0 rpm sample



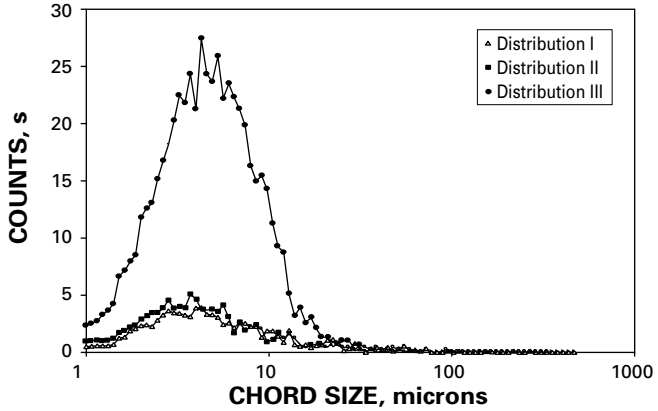
C: Statistics of 1000 rpm sample



D: Distribution of 1000 rpm sample



E: Statistics of 2000 rpm sample



F: Distribution of 2000 rpm sample

6. Statistics and distributions.

their quantity. At the same time, the middle size particles flocculate producing the large size range. Finally the largest size range particles flocculate generating even bigger flocs, with chords greater than 25 µm. The increased number of intermediate and large chords caused the rise of the mean, but this growth was somehow restrained by the increase of the number of small chords—the mean only increased by 30% while an increase of almost 60% was obtained in the sample without centrifugation. Very little displacement of the curve was obtained when observing the chord distributions in the case of 1000 rpm (see Fig. 6D).

For the sample centrifuged at 2000 rpm, the same effect is present in even a more pronounced manner (see Fig. 6E). At the beginning of the experiment, for low PEI doses, the mean experienced a small rise. This was the result of a slight, almost imperceptible, decrease of the number of counts of 1 to 6 µm. After this,

the mean value decreased because of the increased the number of chords between 1 and 6 µm being much greater than the resulting increase in chords between 7 and 25 µm. The absence of a rise in the chords bigger than 25 µm further accentuates this effect.

Figure 6E shows the points where the distributions I, II, and III were obtained. Distributions I and III are the initial and the final ones, distribution II is the one for the mean chord's maximum value. The similarity between distributions I and II can be observed in Fig. 6F. The statistical data remaining practically unchanged and the final distribution demonstrating only a slight displacement compared to the initial distribution. The number of chords has increased for all the sizes (although the rise was very small for sizes between 10 and 25 µm, as a difference from the previous case) except for those bigger than 25 µm.

These studies show the importance of DCM in flocculation processes. Papermakers should consider this type of information when choosing the type and dose of flocculant to use. Moreover, the studies show the effectiveness of the FBRM technique and developed methodology for this kind of investigations throughout the papermaking process.

CONCLUSIONS

Flocculation is a complex phenomena influenced by many parameters. The knowledge of polymer interaction with different pulp fractions under changing papermaking conditions, the monitoring of floc evolution under the influence of shear forces, and the understanding of reflocculation processes are each very important aspects of papermaking. These types of knowledge are difficult to achieve with traditional techniques. The research contained in this work demonstrates the possibilities of using the focused beam reflectance measurement to further the understanding of these important flocculation phenomena.

We also developed a methodology to study the data that allow for a fundamental understanding of the flocculation phenomena and its application to papermaking and demonstrated a practical foundation for added control on the wet-end. This work has supports the use of FBRM to evaluate:

- Flocculation of different pulp fractions.
- Identify flocculation mechanisms.
- Study deflocculation and reflocculation.
- Observe DCM destabilization.
- Determine the influence of contaminants on polymer efficiency.
- Optimize polymer dosage. **TJ**

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