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EXPERIMENTAL INVESTIGATION OF THE INFLUENCE OF FIBRE MORPHOLOGY ON THE INTERRELATION OF FLOCCULATION AND NETWORK STRENGTH

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1 ABSTRACT

It is widely accepted and discussed in the literature that fiber morphological parameters like fiber length, fiber curl and fiber flexibility affect flocculation phenomena and the properties of the fiber network in suspension via mechanical entanglement and forces arising at fiber to fiber contact points. The focus of this work is the interrelation of the parameters floc size distribution, network strength and fiber morphology. Pulp samples of different length distribution, fiber curl, kink index and fiber flexibility are evaluated concerning their flocculation tendency and the strength of the fiber network using methods already established in the literature. The simultaneous measurement allows the investigation of the interrelation of these parameters. It is shown that floc size distribution and network strength are highly correlated. Still, depending on the morphological properties fiber curl, kinkindex and fiber flexibility, samples of comparable floc size distribution and different network strength (and vice versa) are evident.

2 INTRODUCTION

Pulp fibers in suspension form a heterogeneous network depending on consistency and pulp fiber morphology. The rheological properties are governed by these parameters and influenced to a great extent by flocs (mass concentrations of fibers) formed within the network. The characteristics of these flocs and the fiber network are determined by the number of fiber to fiber contacts and the forces at these contact points.

The number of such contacts depends on consistency, on pulp fiber coarseness and on fiber length. These parameters were combined by KEREKES ET. AL. [1] in the so called *Crowding Number N*. The forces at the contact points on the other hand were described by KEREKES ET. AL. [1] as electrochemical, surface tension and mechanical forces. Electrochemical forces may affect the system in case of chemical additives but for pulp fibers at headbox consistency it was early stated by MASON [2] that mechanical surface linkage is the major influence. Surface tension can have an effect if air bubbles are trapped within the network. The mechanical forces can be divided in mechanical surface linking or entanglement due to fiber curl, kinks and extensive fibrillation and in elastic fiber bending (MEYER ET. AL. [3]) due to the fact that fibers in the network are restrained and locked in a bent configuration. The forces exerted in the contact points depend on the number of contacts along the fiber, fiber flexibility and the surface roughness governing the coefficient of friction. The topic of rheology of fiber suspensions has been extensively reviewed by KEREKES [4].

Considerable research efforts were devoted to the measurement of flocculation processes and flocs. Several researchers used fast digital cameras and image analytical methods to evaluate structures in flowing suspension in the turbulent state or in decaying turbulence (e.g. BEGHELLO ET. AL. [5], SALMELA ET. AL. [6], YAN ET. AL. [7]). Another approach to characterize the fiber network is measurement of network strength. Several authors used different kinds of rheometer-devices to conduct such measurements (e.g. BENNINGTON ET. AL. [8], DAMANI ET. AL. [9], HORVATH ET. AL. [10]).

In this work methods for the measurement of both properties – flocculation and network strength – are applied on a set of pulp samples. Flocculation tendency and network strength are then correlated with respect to varying pulp morphological parameters, in order to be able to quantify the effect of a certain fibre property on flocculation on the one hand and on network strength on the other.

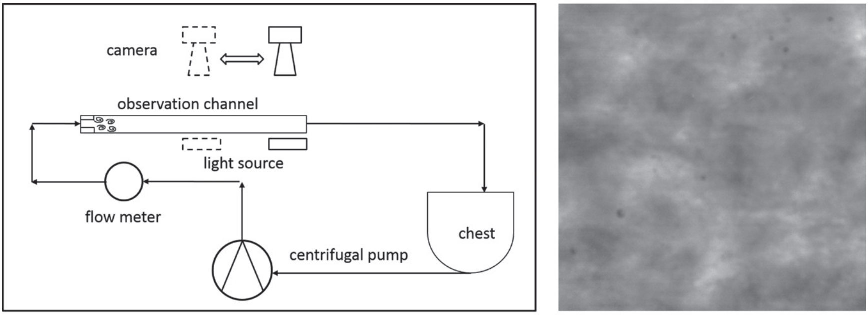
3 EXPERIMENTAL SETUP

3.1 Samples

The samples used for the investigations discussed in the following are a flash dried softwood kraft pulp sample in the unrefined state as well as after refining for 10 and 50 minutes in the valleybeater and an unrefined eucalypt kraft pulp sample. The flash dried softwood kraft sample was chosen because the flash drying process induces considerable curl on the fibers. Therefore the properties of the unrefined sample differ even more from the refined ones if compared to a sheet dried pulp. Every one of these three softwood samples (unrefined and 2 refining intensities) was used to produce mixtures with the eucalypt pulp in ratios 25/75, 50/50 and 75/25. Thereby the fibre length distribution, which is one of the major factors contributing to crowding number, flocculation and network strength, is varied based on the amount of eucalypt fibers. While the properties of the eucalypt sample are constant in all mixtures the effect of refining of the softwood fraction (flexibilisation, reduction of the fiber curl, fibrillation) is accessible based on the specific softwood component mixed with the eucalypt pulp. All measurements are conducted at a consistency of 1%.

3.2 Methods

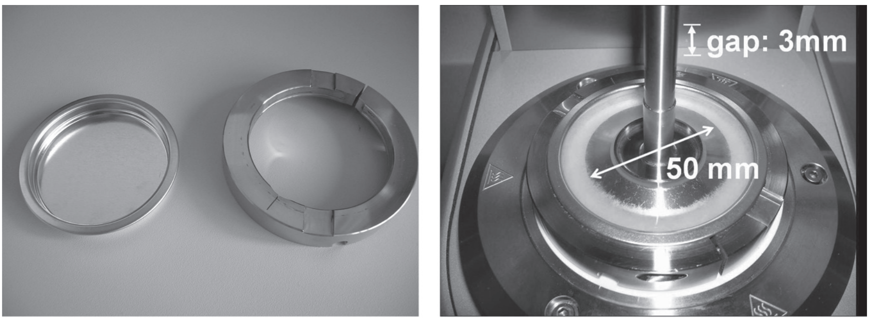
Flocculation: The device used for the measurement of fiber flocculation in flowing suspension is similar to those used by several researchers in the past (Beghello et. al. [5], Salmela et. al. [6]). Pulp is circulated at headbox consistency through a closed loop containing an observation channel of rectangular cross section (35[*mm*] wide, 16[*mm*] in height). The loop consists of a small pulp chest, a centrifugal pump, an inductive flow meter, the observation channel and several meters of polypropylen-tubes. The inlet into the 1[*m*] long observation channel is designed as a constriction block of half the cross section of the channel itself to induce turbulence that decays along the length of the channel. The average flow velocity in the channel for the described measurements is 2[*m/s*] (4[*m/s*] in the constriction block). For the described investigation image acquisition is done 50[*cm*] downstream the constriction block. Previous experiments have shown, that at this point turbulence has decayed and structures/flocs are not subjected to further changes along the observation channel. The flow loop is shown in Fig. 1a. A high speed camera capable of 500 images per second is used to acquire images of the flowing suspension in the observation channel. Illumination is done based on transmitted light using a LED panel controlled by the camera signal. An exemplary image of the flowing suspension as it is acquired with the system is shown in Fig. 1b.



(a) (b)

Figure 1. (a) Schematic sketch of the system for investigation of flocculation phenomena in flowing suspension. (b) An exemplary image of the flowing suspension.

Network strength: Measurement of network strength is performed using a Paar Physica MCR 301 rheometer in a plate to plate configuration similar to what other researchers have used (i.e. Horvath et. al. [10]). The whole setup of the system is standard equipment of the supplier. The measuring head is a so called *CP50-1* with a diameter of 50[*mm*]. The jar containing the suspension resembles the lid of a standard tin can, is normally used as a single-use item for measurements on hardening liquids (see Fig. 2a) and is fixed in a ring mounted on the rheometer. The setup during measurement is shown in Fig. 2b. As several



(a) (b)

Figure 2. (a) The jar containing the liquid during measurement and the ring used to mount it on the Rheometer. (b) The rheometer setup during measurement in the plate to plate configuration.

researchers have found before and is also pointed out by Kerekes [4] in his review, network strength rises to the power of three with increasing consistency. Therefore great importance is attached to sample preparation as only 30[*g*] of suspension are used during one measurement and sampling at 1% consistency at a certain flocculated state is a source of considerable errors. For the evaluation of one sample three measurements are conducted and averaged.

The fiber supension in the gap of 3[*mm*] between the measuring head and the bottom of the jar is deformed elastically during oscillating measurements of small dynamic strain amplitude (0.05) and a frequency sweep of 10 to 1 [*hertz*] at a stepwidth of one. The measured parameter is the so called storage modulus G representing the energy stored to keep a given floc/structure intact and thus representing a measure for the strength of these structures.

Pulp fiber morphology: To be able to relate flocculation and network strength to morphological properties of the fiber material these properties have to be measured as well. These measurements are performed using a Techpap morFi Fiber and Shive analyzer. Although a wide variety of parameters is available with this device only fiber length, fiber curl/kinkindex and fibrillation shall be adressed in this work as these are of obvious interest in context with flocculation and network strength via their effect on mechanical entanglement. A parameter called *bendability* representing fiber flexibility – which is also of high importance when it comes to forces at fiber contact points – is at hand and would be of high interest as fiber flexibility is changed to a great extent during refining in the valley beater and has also considerable effect on flocculation and network strength. Unfortunately this parameter does not deliver meaningfull results. Fiber flexibility seems to be decreased during refining according to the measurement which is not possible. The reason for this flawed result is most probably the measurement principle itself. The *bendability* represents the difference in average curl at two different flow rates in the flow cell. At the higher flow rate the fibers are straightened due to higher shear forces. This effect should be even more pronounced for more flexible fibers. In our case the unrefined fibers exhibit a very high curl due to the flash drying procedure, the refined ones on the other hand show considerably lower curl (see also Fig.5). Thereby the refined fibers allow less difference in curl at different shear rates and in succession deliver lower bendability values.

4 RESULTS AND DISCUSSION

After a first image analytical step to compensate for uneven illumination, structure analysis algorithms (Fast Fourier Transforms) are used on a set of 100 images to determine the floc size distribution in the suspension. Fig. 3 shows the variance distribution over the wavelength (the structure sizes) for the eucalypt pulp and the three softwood samples (unrefined, 10 and 50 minutes in the valley beater). The variance distribution represents the intensity of the structure size in the images. If a certain structure size – a certain wavelength in structure analysis – is very prominent, the corresponding values in the variance distribution are high.

The difference in flocculation between the pure samples is evident in these variance distributions: the unrefined softwood sample clearly shows the strongest flocculation (highest variance in the higher wavelengths/structure sizes); refining reduces flocculation due to flexibilisation, reduction of fiber curl and – for the higher refining intensity – shortening of the fibers; the eucalypt sample shows the lowest flocculation tendency.

The measurement of the storage modulus on the pure pulp samples delivers a similar result with the unrefined softwood sample exhibiting the highest and the hardwood sample the lowest storage modulus and hereby network strength. The mixtures of softwood and hardwood samples show results between the corresponding pure samples. As the main goal of this work was to take a closer look at the interrelation of flocculation, network strength and pulp fiber morphology a direct correlation between flocculation and network strength is necessary. Therefore the

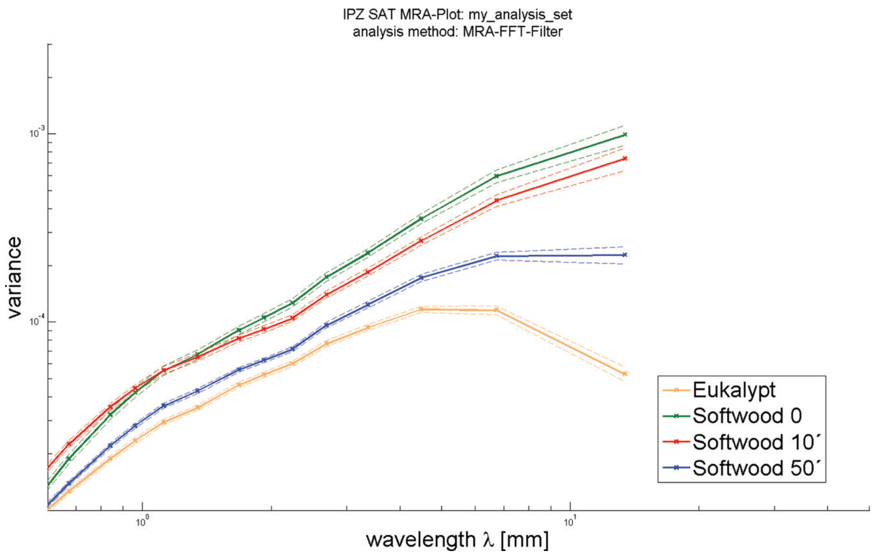


Figure 3. Variance distribution over wavelength for the structure analysis on the pure samples. The dashed lines close to the variance distributions represent confidence intervals based on the evaluation of 100 images.

variance distribution resulting from structure analysis is used to calculate a single value representing the flocculation intensity (integration of the variance distribution over the wavelengths from 0.5 to 15[*mm*]) and correlated to the storage modulus measured with the rheometer. The respective correlation is shown in Fig. 4.

The corresponding fiber morphological parameters are shown in Fig. 5. No absolute values are shown but the relation between the pulp samples for all relevant parameters within one diagram. The fiber length of the softwood sample remains almost constant after 10 minutes in the valley beater and is significantly decreased after 50 minutes. Kink and curl index are rather high for the unrefined sample, mainly due to the flash drying procedure, and are considerably reduced after 10 and even further after 50 minutes due to flexibilisation and straightening of the fibers. Depending on the definition of curl and kink measurement these parameters can be closely related as it is the case in these measurements. The softwood sample shows increased fibrillation due to the increased mechanical treatment in the valley beater. The decreasing flocculation tendency of the refined softwood pulp shown in Fig. 3 can mainly be attributed to these changes in fiber morphology as the decreased curl and kink leads to less mechanical entanglement

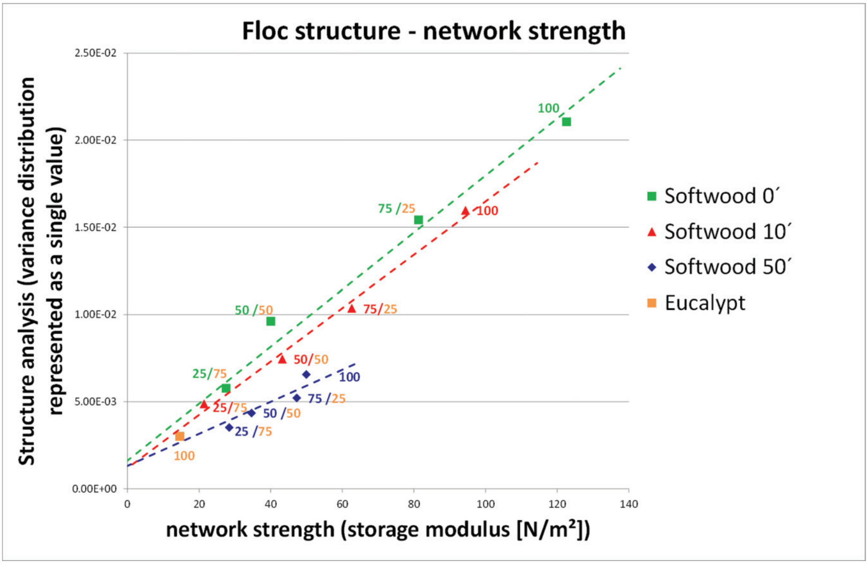


Figure 4. Correlation of floc structure and storage modulus for the pure pulp samples and the mixtures of softwood and eucalypt in different ratios. The numbers beside the data points give the ratio whith the color of the numbers referring to the respective pulp component (see assigned color in the legend).

and the flexibilisation reduces the forces at the fiber contact points. The increased fibrillation might increase the forces at the contact points to some extent, but this seems to be an inferior effect. The eucalypt pulp sample shows lower flocculation tendency due to considerably shorter fibers – a lower crowding number – compared to the softwood sample.

As is evident in the diagram shown in Fig. 4 there is a high linear correlation of flocculation tendency and storage modulus for a given softwood sample and its mixtures with the eucalypt pulp. The added amount of eucalypt pulp decreases the crowding number and therefore leads to a decrease in flocculation as well as network strength. This effect is evident for all three refining levels of the softwood sample and its mixtures with the eucalypt pulp.

The morphological properties of the softwood sample affect the flocculation tendency and network strength depending on the beating level. The decrease in kinks and fibercurl reduces mechanical entanglement considerably and elastic fiber bending is reduced as well due to higher elasticity of the beaten fibers. Other than the changes due to addition of hardwood fibers the changes in the softwood fiber morphology affect network strength and flocculation to a different degree. As can be deduced from the diagram in Fig. 4 pulps with comparable flocculation tendency can show different network strength (and vice versa) depending on the fiber morphological properties.

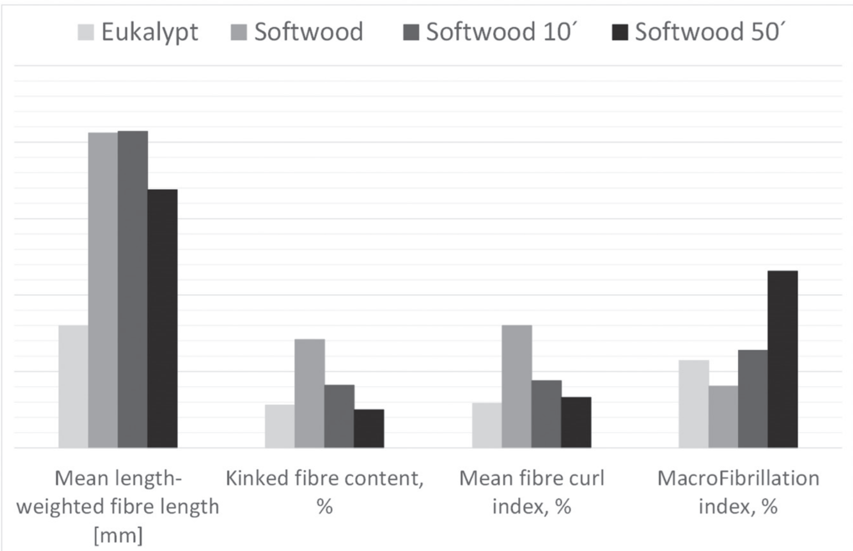


Figure 5. Morphological parameters of the pure pulp samples.

5 CONCLUSIONS AND OUTLOOK

The experiments confirm the qualitative interrelation of morphological and network properties mentioned in the literature. A decrease in fiber length, reduced fiber curl and kink and increased flexibility reduce flocculation and network strength. Based on the parallel measurement of both, flocculation and network strength on the given pulp samples it can be deduced that fiber length has a rather straightforward effect on flocculation and network strength whereas the morphological properties governing linkage and entanglement (kinks, curl) and elastic fiber bending (fiber stiffness) have differing influence on these network properties. Pulps can obviously have similar flocculation tendency and different network strength (and vice versa) depending on morphological properties.

To be able to quantify the influence of certain fiber morphological properties, investigations on a wider range of pulps of different origin and raw material would be necessary.

When it comes to sheet forming and the destruction of flocs to achieve good formation of the final paper not only the size distribution of emerging flocs and network structures is of interest, but also the shear strength of these structures. The combined measurement of both, floc size distribution and network strength, with respect to morphological parameters can be of interest in this context as well.

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Transcription of Discussion

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Warren Batchelor Monash University

I've got one comment, which is that it is not fibre length, but aspect ratio, which controls the flocculation and so when you have a long fibre, you get higher flocculation than for a short fibre, but this is because the longer fibre has a higher aspect ratio. Fibre length is just a shortcut, when you should be talking about aspect ratio.

Matthias Trimmel

Thank you for your comment.

Alessandra Gerli Nalco

Thank you for this very nice presentation. Because you can offset the relationship between network strengths and flocculation, have you looked at the drainage behaviour of suspensions? I can imagine that if you have different floc structure, maybe the drainage will be different. This could provide some useful information for the paper makers.

Discussion

Matthias Trimmel

We did not measure this, but we should do it in the future of course.

Alessandra Gerli

If you are also planning to measure drainage and to look at the drainage behaviour, can you use the freeness of the pulp suspension or do you need a different technique? I can imagine, if you change the aggregate structure, you will get information relating to your measurement technique and maybe you will find out something extra.

Matthias Trimmel

Of course, this is work that I need to do.

Bob Pelton McMaster University

I have never worked in this area other than many years ago we were studying flotation de-inking and we were looking at bubbles moving up through flocculated pulp suspensions and trying to model that. I first saw this type of flow measurement back in the mid '70s, done, by Kerekes and co-workers[3], and I guess that what has changed since then is that the instrumentation is better. What certainly has changed is that we can measure fibre property distributions today in great detail. My question is when are we going to stop doing measurements and just come up with a useful theory? A predictive theory or an equation of state where we can look at fibre properties and calculate the floc suspension properties? I guess this is a challenge to your community as a whole.

Juha Salmela VTT

Very nice to see someone is still working with flocculation, and very nice work. Why did you choose a rectangular channel?

Matthias Trimmel

Because it is easier to construct, and also, if we want to acquire images with a high speed camera, it will be easier to acquire these images in a rectangular channel then in a tube.

[3] R.J Kerekes, R.M. Soszynski and P.A. Tam Doo, "The flocculation of pulp fibres". In Papermaking Raw Materials, *Trans. VIIIth Fund. Res. Symp. Oxford, 1985* (V. Punton, ed.), pp 265–310, FRC, Manchester, 2003. ISBN: 0 9541126 8 7.

Juha Salmela

Yes, but there are, as you probably know, a couple of bad things with a rectangular channel. Its flow field is actually quite complicated because you have vortices in the corners and it is very difficult, for example, to define the local energy feed that you put into the flow at the sudden pipe expansion. Also, you did not show any floc size evolution. It is the saturation floc size that you are measuring, I think, which is not transient any more, but that level has been shown to be very sensitive to the local energy you put into the flow. Do you have a comment about that? Also it would be very interesting to see the floc evolution curve, can you describe it?

Matthias Trimmel

Yes, we have taken measurements over the entire length of the channel from the inlet to the outlet and, after 50 cm, we cannot see any changes, so we have chosen the point 50 cm below the inlet for acquiring images. I don’t have the details of this evaluation with me.

Patrick Huber CTP

I have a comment and then a question. Using your refining procedure, it seems that you had several morphological parameters, like curl and fibrillation, which varied together, so it may be difficult to draw conclusions from the results. Possibly you could study the effect of different types of refiner on those parameters to allow you to vary them more independently. And then the question is: what do you think will happen to flocculation and network strength, when we get to the approach flow and put retention aids in?

Matthias Trimmel

If we add a retention aid, we cannot see the same correlation between floc size distribution and network strength that we see with pure samples. This is one of the reasons why, in the future, we need to measure the network strength by the critical strength and not just by storage modulus. With the method I presented, we cannot give you an answer about the influence of retention aids on the system.

Ramin Farnood University of Toronto

You used transmission images to characterize the state of flocculation of your suspension, and then used a spectral analysis to analyse it. Following that, you used a threshold and integrated the variance from 0.5 to 15 mm. Based on my presentation on Monday, you can avoid some of these steps by using a simple disc model, as I suggested. I am not saying that you should not do a spectral analysis, however a disc model can give you the opportunity to actually extract a single figure for the floc size, instead of integrated variance. With this tool you can look at the evolution of the floc size and the state of flocculation under various conditions.

Jean-Claude Roux Grenoble Institute of Technology-Pagora (from the chair)

I have a question. You characterised the flocculation state and you developed a new index. Did you try to find a correlation with the crowding factor that you also mentioned as characterising the state of flocculation of the pulp suspension? The crowding factor, as you know, is also related to formation number and similar measures and so it is a useful tool. What does your new tool bring to this field?

Matthias Trimmel

As we measure the morphological properties, we also measured the coarseness and the crowding number, which is just influenced by the fibre length, the concentration and the coarseness. And all of our measurements are done at a consistency of 1%, so the concentration is constant and the coarseness of the sample we measured will be similarly constant. And so the crowding number is only influenced by the fibre length.

Jean-Claude Roux

As you know, the aspect ratio is present in the formula:

$$\textit{Crowding Number} = \frac{2}{3} c_v \frac{L^2}{d^2}$$

where C_v is consistency, L is mean fibre length and d is mean fibre diameter.

Matthias Trimmel

In our investigation, we have changed the amount of the eucalyptus pulp and so we changed the length to width ratio in the sample and, with that, we demonstrated the influence of the length to width ratio.

Lars Wågberg KTH

Just a short comment. When you are comparing the long fibre to the eucalyptus, you should also consider the number concentration which is totally different. Eucalyptus pulp has around 25 million fibres per gram, whereas a long fibre has around 2.5 million. So I do not know if it comes out of the fibre wall thickness, but it's a big difference between the fibres.

Torbjörn Wahlström Stora Enso

It's good when there are a lot of comments so that you have time to figure out a good question. You started with different furnishes, different refining, etc., then you started to think as a paper maker: how to use this for paper making? You mentioned in your conclusions that the results can also be of interest with respect to formation of the final paper. How do you use these results to get to that? What is your next step in this direction? How will we use this?

Matthias Trimmel

In our next step, and this will be my main work, we will extend this circulation loop with a little head box and a wire and we will try to measure retention, dewatering and also formation. We want to compare the results we got in this investigation with the results we hope to get with the new equipment. Then I can tell you more about the influence of flocculation and network strength on formation.