A METHOD CAPABLE TO DETERMINE DAMAGE OF THE OUTER FIBER WALL LAYERS

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Abstract

Cupri(II)ethylendiamin is normally used to dissolve chemical pulp fibers. If used in a defined concentration and for restricted reaction time it only provokes heavy fiber swelling. In areas where the outer layers of the fiber are too weak due to mechanical or chemical influence, they can no longer restrict this swelling process - they break and strong swelling reactions can be observed under the microscope.
Images of the swollen fibers are acquired and digital image analysis is used for evaluation of the swelling intensity. For each individual fiber a so called “degree of swelling”, representing the swollen proportion of total fiber length, is evaluated. This degree of swelling corresponds to the damage of the outer fiber wall layers.

I. Introduction

This paper presents a novel method capable of delivering information regarding the condition of the outer fiber wall layers of chemical pulp fibers.
The cell wall is composed of two layers, the thin primary wall (P) and the thick secondary wall. The secondary wall is divided into the three sublayers S1, S2 and S3. The orientation of the microfibrils differs between these layers. The fibrils in P are aggregated in a rather stochastic way, the S1 layer has a crossed fibrillar structure and the fibrils within the S2 are highly aligned. Therefore the S2 layer shows the highest swelling ability.
As already mentioned our method is based on chemically induced swelling of chemical pulp fibers. In areas where the more complex structured S1 layer is weakened or damaged during the pulping or stock preparation process, it can not restrict the chemically induced swelling of the S2 layer and characteristical swelling reactions can be observed (**Figure 1**).
The quantity of such swelling reactions reflects the condition of the outer fiber wall layers. Several methods described in the literature are based on this principle.
Brecht and Nisser [1] used cupri(II)ethylenediamine to assess fiber wall damage. Hortling et al. [2] use iron-sodium-tartrate (EWNN) and Unger et al. [3] quantitatively evaluate swelling and dissolving of pulp fibers in EWNN and LiCl/DiMAc (lithium-chlorine/dimethylacetamide). Ander and Geoffrey [4] indicate, that polarized light microscopy, electron microscopy and fiber swelling induced by chemicals indeed all indicate cracks in the S1 fiber wall.

II. Experimental

Like Brecht and Nisser [2] we use cupri(II)ethylendiamin as the swelling chemical. Different to the methods mentioned above, we do not observe the swelling reaction itself, but we asses the fibers after treatment.

The swelling reaction is started by adding diluted cupri(II)ethylendiamin solution to 20g of suspension (1g/l). After 20 sec the reaction is stopped by dilution with deionised water.
A CMOS-camera with a resolution of 6,25 µm/Pixel is used to acquire images of the treated fibers by means of transmitted light microscopy in a prototype flow cell. For the evaluation of one pulp sample 1500 images containing about 2500 objects with a minimum length of 250 µm are taken. The time need for image acquisition in the prototype is about 45 minutes.
Figure 1 shows treated fibers; some with the typical swelling reactions – volume swelling, gel swelling, balloon swelling – indicating damage of the outer fiber wall layers, with some still completely unswollen.

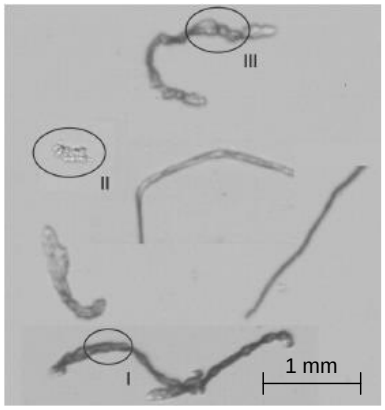

Figure 1. Characteristic types of swelling: Volume swelling (I), gel swelling (II), balloon swelling (III).

Image analysis algorithms developed by Hirn [5] determine the proportion of total fiber length showing the characteristic swelling reactions. Each fiber is assigned a so called "degree of swelling" in % swollen fiber length.
The repeatability of the method was tested on the basis of three measurements on a softwood kraft pulp sample. Sampling, treatment with the chemical, image acquisition and analysis were performed three times independently. The average degree of swelling was 30,3% (swollen fiber length), the standard deviation on the basis of the three measurements was 0,3%.

III. Results and discussion

To give an impression regarding the applicability of the method, some results on topics we have already investigated are presented:

Comparison of pulp samples

The comparison of pulp samples is restricted to similar samples in terms of raw material (hardwood/softwood) and cooking process (kraft/sulphite). It is impossible to compare for example sulphite and kraft pulp samples as the condition of the outer fiber wall layers is totally different due to the different characteristics of the cooking liquor.
As an example we compared six softwood kraft pulp samples of similar brightness between 85 and 90%. The average degree of swelling lay between 23 and 51%, which is comparable to the difference between an unbeaten sample and the same sample after 250kWh/t in an industrial refiner. This shows the enormous differences in the condition and the strength of the outer fiber wall layers that occur even between similar commercial pulp samples.
In the comparison we found a clear correlation between the average degree of swelling and the refining resistance, represented by the number of revolutions in the PFI-mill needed to

reach a degree of beating of 23SR. The correlation is shown in **Figure 2**. As the degree of swelling represents the strength and condition of the S1 layer the interrelation to the refining resistance is quite plausible.

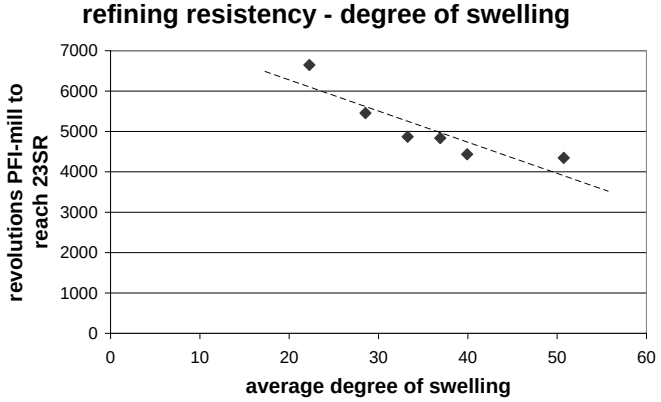

Figure 2. Correlation between the average degree of swelling and the refining resistency for six kraft pulp samples of similar brightness

Refining

The best applicability of the method was found for refining characterisation. As an example we compared three different aggregates: a Jokro-mill, a PFI-mill and a laboratory disc refiner. The comparison was done on the basis of a softwood kraft pulp sample. It was refined to an identical breaking length of 7km with the three laboratory refiners. The degree of beating [SR] was 16 for the Jokro- and the PFI-mill beaten sample and 35 for the laboratory disc-refined one. Both, the Jokro-mill as well as the PFI-mill develop the breaking length rather gently compared to the harsh treatment in the disc refiner. Increased bonding is based more on flexibilisation of the fibers than on fibrillation and fines. The degree of beating as well as the average degree of swelling for the three aggregates is shown in **Figure 3a**.
The disc refiner shows the highest degree of swelling at the specific breaking length, which means the S1 layer took the highest damage during refining. Comparing the Jokro- and the PFI-mill a difference in the degree of swelling can be observed (27,3% Jokro-mill compared to 31,5% PFI-mill average swollen fiber length).
The PFI-mill causes higher friction on the fiber surface due to the higher speed difference between rotor and housing. In the Jokro-mill the rotor is more or less rolling in the housing and less friction occurs. We have slightly more fibrillation and therefore higher damage of the outer fiber wall layers for the PFI-mill treated pulp sample. This becomes evident in the degree of swelling, although the degree of beating shows barely any difference.

As damage data is available on every single fiber it is possible to assess the homogeneity of the refining process. As an example, **Figure 3b** shows the fiber damage distributions over five classes of a softwood kraft pulp sample, refined with three different industrial aggregates: a double cone refiner, a double disc refiner and a cylindrical refiner.The parameters refining consistency, specific edge load and specific energy consumption were identical for this trial.
The mean calculated damage of the fibers was similar for the double cone and the double disc refiner at 42,6 and 43,0 %. The pulp sample refined with the cylindrical aggregate showed a higher average degree of fiber swelling of 46,6 %.
As can be seen in **Figure 3b**, the sample refined in the cylindrical aggregate exhibits less objects in the class of rather undamaged fibers of 0 % - 20 % swollen fiber length. These fibers either did not reach the refining zone, or they were not damaged, not fibrillated during

treatment. This result indicates that the higher average fiber damage after treatment with the cylindrical aggregate is not due to higher damage of the fibers that have been treated in the refining zone, but due to a higher percentage of damaged fibers.

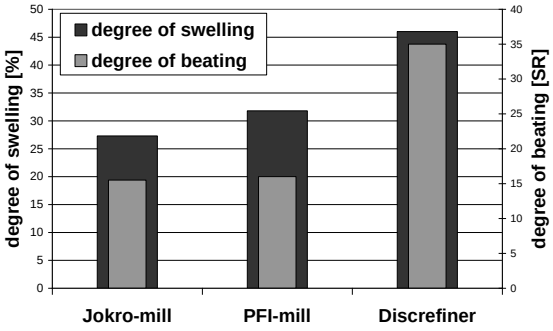

Figure 3a

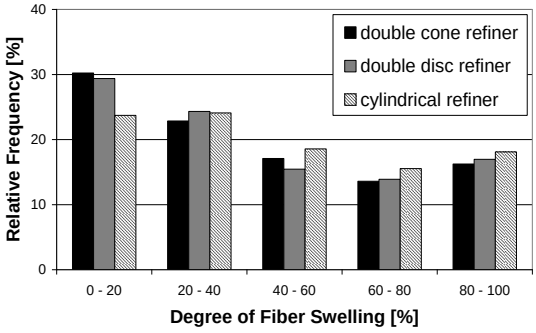

Figure 3b

IV. Conclusions

The described method is capable to determine the condition of the S1 layer. The highest applicability lies in the field of refining characterisation. Combined with data on fiber length development, the method is capable to determine the character of a refining process concerning shortening, fibrillation and flexibilisation. Furthermore, as data is available on a single fiber basis, the method can be used to investigate the homogeneity of the refining process.
Comparison of different pulp samples is possible within equivalent cooking process type, raw material and similar bleaching level and delivers information concerning the refining resistance.

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