A Method to determine Fiber Wall Damage induced by Refining

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Abstract

A chemical provokes heavy swelling of chemical pulp fibers. In areas where the outer layers of the fiber wall were damaged during refining, strong swelling reactions can be observed under the microscope.
Images of a highly diluted suspension containing the swollen fibers are acquired and digital image analysis is used for evaluation of the swelling intensity. The characteristic morphology of swollen fiber regions is detected. For each individual fiber the so called "degree of swelling", representing the swollen proportion of total fiber length, is evaluated. This degree of swelling corresponds to the extent of damage the fiber walls have taken during refining.
As data is available on every single fiber, the method is capable to determine the distribution of fiber wall damage over the pulp sample and therefore assess e.g. the homogeneity of the refining process.

1. Introduction

This paper presents a method that delivers information on fiber wall damage. It might be a meaningful upgrade in the field of pulp characterisation based on single fiber measurement, as an addition to common morphological parameters like fiber length and width.
The cell wall is composed of two layers [1], the thin primary wall (P) and the thick secondary wall. The latter layer is divided into three sublayers S1, S2 and S3. The texture of the cellulose 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 which promotes extensive swelling.
For our method a swelling chemical is used to provoke strong fiber swelling. In areas where the more complex structured S1 is damaged, it can not restrict the chemically induced swelling of the S2 layer and characteristical swelling reactions can be observed (Fig. 1). So the quantity of such swelling reactions reflects damage of the S1 layer. There already exist several other methods which are based on this principle
Brecht and Nisser [2] used cupri(II)ethylenediamine as a swelling chemical to assess fiber wall damage. Correlations between freeness and fiber swelling are reported.
Hortling et al. [3] use iron-sodium-tartrate (EWNN) in order to examine fiber swelling, fibrillation, nodes of damage and curl index by visual inspection of photographs taken at specified time intervals after addition of the chemical. Unger et al. [4] quantitatively evaluate swelling and dissolving of pulp fibers in EWNN and LiCl/DiMAc (lithium-chlorine/dimethylacetamide) by recording video images of single fibers dissolving and applying image analysis to evaluate changes in fiber thickness and fiber transparency. They confirmed the lower swelling reactivity of kraft pulps compared to sulphite pulps, a fact that has been known widely and was also found by our method. Recent work by Ander and Geoffrey [5] proves, that polarized light microscopy, electron microscopy and fiber swelling induced by chemicals indeed all indicate cracks in the S1 fiber wall.

2. Experimental

We adapted the method of Brecht and Nisser [2] where cupri(II)ethylendiamin is used as the swelling chemical. Unlike the methods mentioned above, we do not observe the swelling reaction itself with photographs or video, but we evaluate the swelling intensity of fibers after controlled treatment with the swelling chemical.
A beaker, filled with about 20 g of suspension (consistency 1 g/l) is put on a magnetic stirrer. The swelling reaction is started by adding diluted cupri(II)ethylendiamin solution (pure chemical would lead to immediate dissolution of the fibers) to the suspension. After 20 sec the reaction is stopped by dilution with deionised water.
Images of the treated fibers are taken by means of transmitted light microscopy in a prototype. The highly diluted (consistency 0.02 g/l) suspension is pumped through a transparent flow cell and a CMOS-camera with a resolution of 6,25 µm/Pixel acquires images of size 6,25 x 14,5 mm.

For the evaluation of one pulp sample 1500 images are taken. These images contain on average 2500 objects with a minimum length of 250 µm.
Fig. 1 shows some examples for treated fibers, some of them showing the typical swelling reactions – volume swelling, gel swelling, balloon swelling – indicating damage of the fiber wall, some of them completely unswollen.

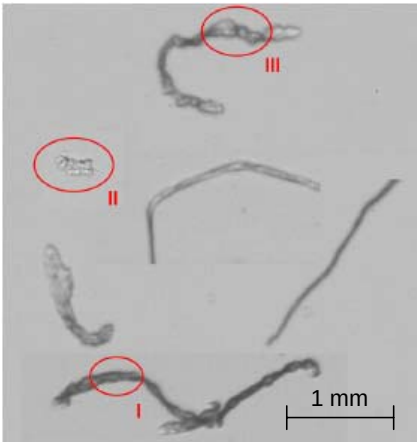

Fig. 1
Characteristic types of swelling: Volume swelling (I), gel swelling (II), balloon swelling (III).

Image analysis algorithms developed at our institute by Hirn [6] are used to determine the degree of swelling for each individual fiber. Fig. 2 shows an exemplary image of a partly swollen fiber.

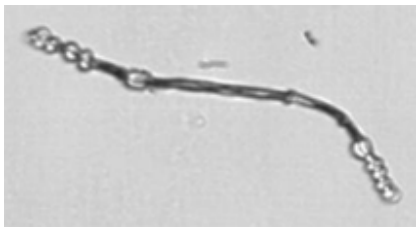

Fig. 2
Fiber with a degree of swelling of 36,4 %, which means that 36,4 % of total fiber length are rated swollen by image analysis

Image analysis determines the proportion of total fiber length which shows the characteristic swelling reactions. In Fig. 2 36,4 % of total fiber length were rated as swollen, therefore a proportion of 36,4 % of the fiber is considered to have a damaged S1 wall.
Fig. 3 shows the repeatability of the method on the basis of three measurements of a softwood kraft pulp. The diagram shows the frequency distribution of the degree of swelling over five classes. 46 % of the fibers show hardly any damage (0 % - 20 % swollen fiber length) whereas for 7 % of the fibers nearly the whole fiber length was identified as swollen by image analysis (80 % - 100 % swollen fiber length).

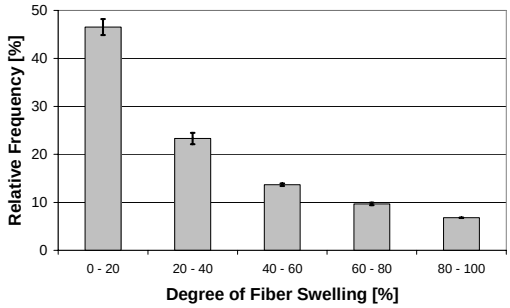

Fig. 3
Repeatability on the basis of three fiber swelling measurements. The error bars represent a 95 % confidence interval.

3. Results

Combining the data on the swelling behaviour with the fiber length allows an extended characterisation of refining processes. The degree of swelling corresponds to outer fibrillation and the fiber length development reflects shortening during refining.
A softwood kraft pulp sample was refined to identical breaking length of 7 km with three different aggregates. The goal was to determine differences concerning the treatment of the pulp between the Jokro-mill, the PFI-mill and a pilot scale disc refiner. The degree of beating for the two laboratory mill-treated samples was similar at about 16 SR, the disc refined sample had a SR-value of about 35.
Fig. 4 shows a contourplot of the degree of swelling versus fiber length. The two-dimensional frequency distribution of fibers is visualized by colour. Bright areas represent numerous fibers, dark ones represent only few or no fibers at all.
Only little difference can be observed between the plots corresponding to the Jokro and the PFI mill. In the Jokro mill plot, there is a higher concentration of fibers in the region of low degrees of swelling, representing rather undamaged fibers.
Comparing the plot corresponding to the disc refiner with those of the two laboratory aggregates, much higher fiber concentrations in the region of high degrees of swelling, representing heavily damaged fibers, are evident. Furthermore a shift of the high fiber concentrations to the left – to shorter

fiber classes – can be observed due to increased shortening of fibers in the disc refiner.

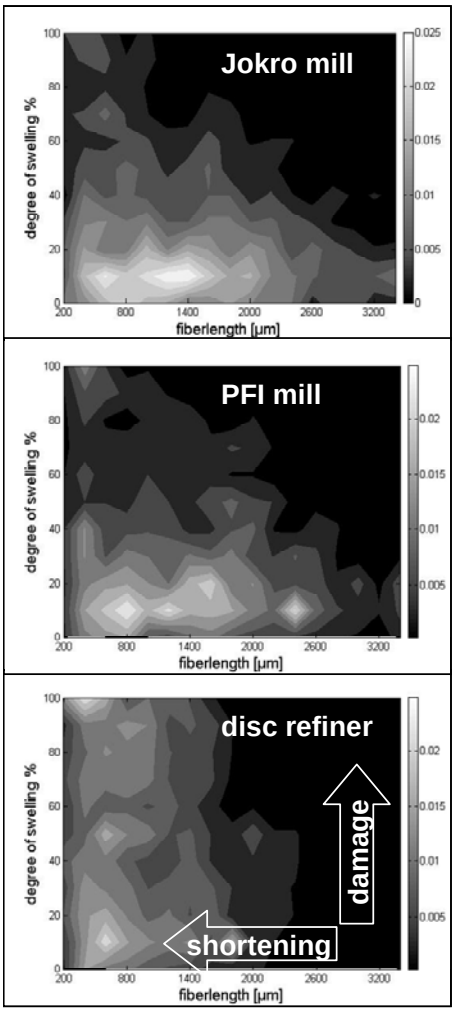

Fig. 4

Considering the same results visualized as frequency distributions of the degree of swelling (Fig. 5), the difference between the Jokro- and the PFI-mill becomes more obvious. Although it can be assumed that all fibers pass the refining zone several times in such laboratory mills, a considerable amount of fibers shows rather no damage of the fiber wall. The PFI-mill treated pulp sample shows less fibers in the class of such undamaged fibers (0 % - 20 % swollen fiber length) than the Jokro-mill. That is due to the fact, that in the PFI-mill there is higher friction between rotor and housing and therefore the refining is more fibrillating. In the Jokro-mill, where the rotor rolls in the housing, breaking length descends to a higher extent from fiber flexibilisation. It is developed smoother and more time consuming. Therefore less damage is imposed on the fiber wall.

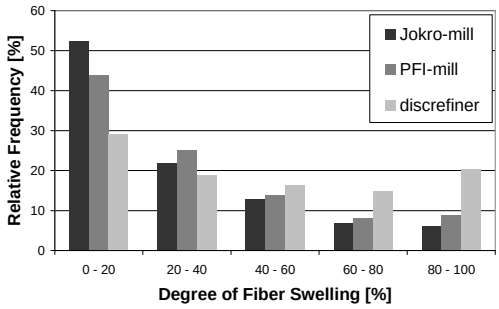

Fig. 5

As damage data is available on every single fiber it is possible to assess the homogeneity of the refining process. As an example, Fig. 6 shows the fiber damage distributions of a softwood kraft pulp sample, refined with three different industrial aggregates: a double cone refiner, a double disc refiner and a cylindrical refiner.

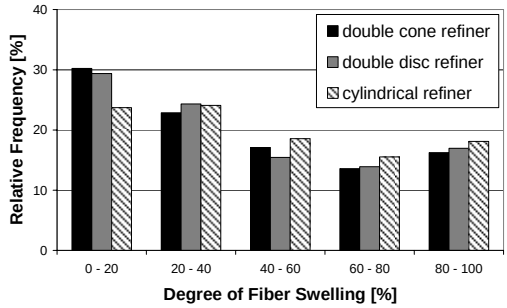

Fig. 6

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 Fig. 6, 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.

The cylindrical refiner treats the pulp suspension in a more homogeneous way, as less fibers remain undamaged compared to the other industrial aggregates.

4. Conclusions

The described method is capable to deliver information concerning the fiber wall damage measured on individual fibers. Together with fiber length measurement it allows an extended evaluation of refining processes.
The fiber wall damage that occurs to reach a certain breaking length can be compared for different refining methods. Different refining concepts and influences like refining parameters or different refiner fillings can be investigated.
Besides, the homogeneity of the refining process can be assessed with this method.

5. Acknowledgements

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