TECHNISCHE UNIVERSITÄT WIEN
Vienna University of Technology

Diploma Thesis

Study on the Grinding Process of Cellulosic Fibers and Characterization of thereby Obtained Short Fibers

Carried out at the

Institute of Chemical Engineering of Vienna University of Technology

under the supervision of

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Abstract

A research project was carried out which on the one hand investigated the fiber grinding process in a cutting mill and on the other hand analyzed the possibilities to determine the fiber length of the obtained fibers. It was within the scope of the project to determine the main steering parameters that affect the obtained fiber length. The relationship between the resulting fiber length with the fiber titer, starting length, different additives and manufacturing methods, the used sieves and the process mode have been determined. It is thus possible to state the milling parameters for a certain fiber type in order to meet a specific fiber length or vice versa to predict the fiber length under certain grinding conditions also for a certain fiber type.

In the second part of the work the ground fibers have been analyzed in terms of fiber length and fiber length distribution. As standard analysis method a commercial fiber analyzer MorFi was used which was originally developed for paper and pulp characterization. To validate the results of the MorFi analyzer an alternative method has been developed. Images have been recorded with a conventional office image scanner at a resolution of 6400 dpi. The key problems turned out to be the image contrast and the proper dispersion. In particular white fiber material results images of low contrast, which can hardly be used for automated image analysis. It was demonstrated that the contrast could be significantly improved by using a reactive dye. Furthermore the optimal technique to obtain a proper fiber dispersion was developed. On the one hand a proper fiber concentration is necessary to prevent overlapping fibers as well as a dispersion agent with a high viscosity to prevent sedimentation effects is essential. The digital images have been analyzed using two different image analysis routines. Both routines and MorFi gave comparable results.

In conclusion, the study determined the key parameters for the fiber grinding process. Furthermore different fiber analyses are proposed which result in precise and repeatable results.

Kurzfassung

Im Rahmen des Forschungsprojekts wurden einerseits Untersuchungen des Faserzerkleinerungsprozesses in einer Schneidmühle und andererseits Analysen der Faserlänge bzw. Faserlängenverteilung der erhaltenen Fasern durchgeführt. In weiterer Folge wurde versucht die Parameter, welche die herstellbare Faserlänge beeinflussen zu ermitteln. Die Einflüsse des Fasertiters, der Schnittlänge, der Additive und der Herstellungsmethode der verwendeten Fasern sowie des verwendeten Siebes und der Prozessfahrweise konnten bestimmt werden. Dadurch ist es möglich die Mahlparameter für eine bestimmte Faserlänge bei Einsatz eines bestimmten Fasertyps festzulegen oder umgekehrt die Faserlänge unter bestimmten Mahlbedingungen bzw. für einen bestimmten Fasertyp vorherzusagen.

Der zweite Teil der Arbeit befasste sich mit der Charakterisierung der gemahlenen Fasern hinsichtlich Faserlänge und Faserlängenverteilung. Als Standardanalysenmethode wurde der kommerzielle Faseranalysator MorFi verwendet, welcher eigentlich für die Papier- und Zellstoffcharakterisierung entwickelt wurde. Um die Ergebnisse des MorFi Analysators validieren zu können, wurde eine Alternativmethode entwickelt. Mit einem konventionellen Büroscanner, welcher eine physikalische Auflösung von 6400 dpi hatte, wurden digitale Bilder der Faserproben angefertigt. Als Hauptprobleme erwiesen sich der geringe Kontrast, sowie die Dispergierung der Fasern. Besonders bei weißem Fasermaterial war der Kontrast zu gering und eine zufriedenstellende Bildanalyse kaum möglich. Durch Verwendung eines Reaktivfarbstoffes konnte der Kontrast signifikant verbessert werden. Des Weiteren wurde einen Methode zur optimalen Faserdispergierung gefunden. Hierfür ist die richtige Faserkonzentration wichtig, um ein Überlappungen einzelner Fasern zu verhindern. Außerdem muss dafür ein Dispersionsmittel verwendet werden, welches durch eine hohe Viskosität Sedimentationseffekte unterbindet. Die angefertigten Bilder wurden schließlich mit zwei unterschiedlichen Bildanalyseprogrammen (Scientific Counter und AnalySIS) ausgewertet und mit den Analyseergebnissen von MorFi verglichen. Dabei wurden vergleichbare Ergebnisse erzielt.

Es wurden die relevanten Mahlparameter für den Faserzerkleinerungsprozess bestimmt und eine Faseranalysenmethode entwickelt, welche präzise und reproduzierbare Ergebnisse liefert.

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1 Motivation

Grinding in a cutting mill is still a quite unexplored process. Hence an important focus of this project was to determine the main steering parameters that affect the obtained fiber length. It was assumed that the fiber titer, starting length, different additives and manufacturing methods of the examined fibers, the used sieves and the process mode influence the resulting fiber length in the grinding process. Due to these assumptions a series of experiments was carried out to examine these influences.
It is not only important to know which process parameters affect the resulting fiber length and how it is influenced. The second focus of the work was to analyze and characterize the produced ground fibers concerning their length and length distribution. Because of this fact in the second part of the work a cheap and easy characterization method for fiber length and length distribution has been developed. The validation of the actual standard characterization method, which is used at the institute of chemical engineering, was also part of this work.

2 Theoretical Background Particle Characterization

Characterization of particles became quite fast and easy in the last years. Quick and accurate tools for determination of size, size distribution and also shape are commercially available. Most of these tools have been developed for rounded particles, but fibrous particles often cause problems. For nearly every chemical process it is necessary to gain information about the used or produced particles. It is the same with fibers. Fibers represent a disperse system, which consists of innumerable individual particles [1]. Particulate systems are normally characterized according to certain attributes. Especially size and shape are of importance. Naturally particles are irregularly shaped. Hence methods are established to calculate statistic diameters like the Feret or the Martin diameter [2-5]. For describing fibers the particle size is not a very useful tool because length and width are quite different [1]. In the literature no clear definition of what a fiber is can be found. In textile industry, the biggest and most important user of fibers, a fiber is defined as a linear particle, which can be processed to textiles [6]. The fiber producing industry gives a more far-reaching definition. Following this definition a fiber is a morphological term for

substances characterized by their flexibility, fineness and high ratio of length to cross sectional area. Based on this definition it is possible to subdivide fibers into fiber fly or airborne fibers, flock, which are very short fibers for other purposes than spinning, staple fibers which are of limited but spin-able length and filaments, whose length is considered as continuous [7]. In the last years several methods for analyzing fibers have been developed, also automated fiber analyzers. They are used in pulp and paper industry and are commercially available [8]. Bartl et al. also demonstrated that these characterization methods are applicable for short fibers [9].
In the following chapters a few possible methods for fiber analysis will be introduced.

2.1 Laser Diffraction Particle Size Analysis

A very common way of particle size characterization is laser diffraction. It is well established today, as it is a quick and reliable method. Particles passing through a laser beam scatter light at an angle directly related to their size. Interpreting this data makes it possible to calculate particle size distribution. There are two possible methods for interpreting the data, the Fraunhofer and the Mie theory. Based on the interpretation method the particle size may range from about 0.02 µm to 2000 µm. With this system it is only possible to get information about size but not about shape. This fact makes this system not convenient for analyzing fibers. Bartl et al. showed in their work that laser diffraction is not applicable for samples, which significantly exhibit a large deviation between length and width [1].
Berthold et al. used a Laser Diffraction system for analyzing fiber collectives by modifying the detector unit of the device. Their idea based on the fact that in many commercially available laser diffractometers a laminar flow of the suspension medium in the measurement cell exists. Data analysis carried out using commercially available methods is normally based on the assumption that there is a statistical orientation of the particles in the measuring cell. Received diffraction patterns are assumed to be centrosymmetric and ring shaped. Hence the detectors in commonly used machines only record parts of the diffraction pattern of a fibrous particle. As a result, grain size analysis of fibrous particles gives an equivalent diameter between length and diameter. Berthold et al. showed that fibers align in the flow direction when using a Malvern Mastersizer X. They estimated that the entire diffraction pattern should provide information about length and diameter of the fibers. Berthold

et al. modified the Mastersizer X in a way that it was possible to rotate the detector unit along the optical axis. This made it possible to measure the intensity distribution of the diffraction pattern under different azimuthal angles. They measured wollastonite fibers at azimuthal angles of 0°, 45° and 90°. The fiber collective showed the estimated behavior. The measured grain size increased with increasing azimuthal angle of the detector position. In the 0° position mainly the fiber diameter was measured and in the 90° position the length. Berthold et al. suggested that it is possible to distinguish isometric particles [10].

Several methods have been described for determining the aspect ratio of fibers using light scattering techniques. However most of these methods are designed to determine the aspect ratio of single fibers. Characterizing fiber collectives is not possible [11-25].

Only Heffels et al. managed it to develop a set-up to characterize the aspect ratio of fiber collectives from the azimuth intensity fluctuation of the diffraction pattern and the use of statistical correlations to reconstruct the single particle diffraction pattern. The diffraction pattern of a particle collective is therefore a linear combination of the single particle patterns [15, 20]. Because of that fact, it is clear that the shape information gets lost with an increasing number of particles. Heffels et al. claimed that the number of particles in the laser beam might vary from 10 for octagons to about 80 for fibrous particles. In practice, the maximum possible amount of particles is 20 overall, depending on the shape [15]. Hence his method also does not allow a suitable characterization of particle collectives.

Until now, no commercially available fiber analyzer based on laser diffraction is available.

2.2 Optical Microscopy, Automated Image Analysis and Skeletonization Process

Size and shape of particles can be determined quite simply by using light optical microscopy. It is very accurate, but time consuming. The interpretation of the recorded images is frequently not automatized or only semi automatized. BGI recommended optical microscopy for the determination of airborne fibers in 2004. The problem is that for gaining statistically valid data, at least 100 individuals have to be evaluated. This is a very time consuming process [26]. Optical microscopy

software systems are able to create quite large images, which are constructed from several small images. This makes it possible to use a high magnification in order to evaluate for example fiber width. On the other hand it is also possible to determine the fiber length, even of long fibers around 2 mm, which are covered by more than one image [1]. The combination of optical microscopy with modern microscopy software systems allows a fiber characterization with high accuracy [26]. Quick and economic determinations are not possible with these systems [1].

Digital image analysis technology has been developed rapidly in the last few years. So a multitude of new applications in the field of fiber assessment have been developed. Most fiber analysis methods based on image analysis are systems in which the fibers are suspended in a fluid. They are photographed in a flow cell and then their properties are evaluated by digital image analysis [8]. A very simple process in image analysis is the separation of dark and light pixels of an image. The user defines the areas of the image to be considered and the parameters to be calculated (e.g.: area, perimeter, etc.). The software will compute the parameters, which comply the given criteria. This system makes it possible to measure for example fibers, which are lying on a diaphanous material and are illuminated with light. Several digital imaging software systems operate that way.

For commercial analysis more and more automated fiber analyzers are available. An important group of these systems are flow-cell based systems. The fibers are measured in a suspension, which is pumped through a flow-cell. The combination of a flow-cell with an image analysis system is a very efficient concept for the characterization of fibers. The measurement can be carried out in a fully automated manner. If the hard- and software components of these systems are stable enough, they can be used for online process control, e.g. in the pulp industry [8].

In the following a few commercially available systems will be envisaged. All of them are based on a rear illumination flow cell and an image analysis system [8].

The Galai CIS-100, which is also called Pulp Fibre Analyzer, has got a pulsed flash as a flow cell light source. The images are recorded with a CCD camera. From every pulp sample, around 2000 fibers are analyzed in terms of length, width and curl [27].

The FiberLab produced by Metso has got two light sources and three optical detectors. This setup makes it possible to perform a wide variety of fiber measurements. A low-resolution camera provides images for the determination of

fiber length, coarseness, kink and curl. A second, high-resolution camera measures fiber width, cell wall thickness and cross sectional areas. A light scattering detector can measure smaller particles [28].

A two-camera system called Fibermaster has been developed by STFI. It provides fine and coarse resolution images. Due to hard-wired image processing as camera on-chip, measurements are considerably faster than other systems. The system can measure length, width, coarseness, kink and also two shape factors for curl. It can identify and count vessel cells and the shive content is evaluated too. This system is the only one that can determine fiber flexibility. Shear forces increase with flow speed in the flow channel. The Fibermaster System measures the change of fiber curl at different flow rates in the measurement cell. Large changes of fiber curl upon changes in flow speed indicate high fiber flexibility. So fiber flexibility is defined as the change of fiber curl at different flow rates [29].

FQA sells its OpTest Fiber Quality Analyzer. It has got a special flow cell design to prevent fouling and the formation of deposits. Three currents flow through the cell, only the middle current contains fibers, the others, flowing next to the wall, consist of pure water. In this case a circular polarized laser flashlight provides transmitted illumination. Other flow cells work with linear polarized light. Length, width, coarseness, kink and curl can be measured with this system [30].

Since the 1980ies, the Kajaani FS-200 is on the market [31]. It has been widely used since. It follows a totally different working principle than other common systems. The sensor is a line camera, which is assembled along the length axis of a capillary, which carries the fiber suspension. Transmitting polarized light illuminates the capillary. When fibers pass the sensor, they induce changes in the polarization of the light, which are detected by the sensor. Hence the 1 – dimensional projection of a fiber is measured. As a result only length and coarseness can be determined. With its rather low resolution of 50 µm/pixel fines measurement is only possible to a limited extent [28].

In this research project the MorFi fiber analyzer from Techpap was used. It is equipped with one high-resolution camera. It is possible to determine fiber length, width, coarseness, kink and curl. Fiber fibrillation can also be determined. It is defined as the length of all micro fibrils attached to the fibers in relation to total fiber length [32]. Aim of this work was also to compare the results for fiber length with the new developed methods.

A very important digital process that is carried out by the MorFi software and also by a lot of other image analysis tools is the skeletonization process. This is a way to reduce dimensionality of digital objects and is of interest in a number of tasks for image analysis. The aim of this process is to extract the medial representation of a digital object with lower dimensions. Algorithms that process the same way have been called thinning and medial axis transformation. The resulting set was called medial line, medial axis transformation, skeleton or labeled skeleton. These medial representations don't have to affirm and also show different qualities. Skeletonization is a process to create a subset of the object, the skeleton, which is topologically homotopic to the object, spatially placed along the medial region of the object and should account for different morphological and geometrical properties of the represented object. The skeleton is the starting point for the reconstruction for the parts of interest of a digital object. At the beginning skeletonization has been used to compute linear representations of 2D digital objects. In the process object pixels on the border of the object are changed to background pixels until the "skeleton" is obtained. 2D Skeletonization has been largely influenced by the work of Blum. He worked with geometry based on the primitive notions of a symmetric point and a growth process [33, 34]. Skeletonization also works in 3D. The basic idea is the same; object voxels are changed into background voxels, provided that topology and geometry are not altered until the skeleton is obtained [35].
The MorFi software from TechPap uses this technique and it was assumed that it helps towards separating striking fibers or insufficiently dispersed fibers, but also to analyze the fiber surface.

2.3 Rectangle Model according to Beck

To find out if the MorFi system produces reliable results, it was necessary to find a method to characterize fiber collectives in reference to their fiber length distribution. This method for determining the length of short fibers is based on the rectangle model, which was introduced by Beck [36]. A fiber can be, in good approximation, described as a rectangle. Most microscopy software systems are able to detect the area and perimeter of an object. With these two parameters, length and width can be calculated. Equation 1 gives the fiber length, Equation 2 the width.

$$l = \frac{P}{4} + \sqrt{\left(\frac{P}{4}\right)^2 - A}$$

$l \ldots\ldots\ldots\ldots \text{length}\ [\mu m]$

$p \ldots\ldots\ldots\ldots \text{perimeter}\ [\mu m]$

$A \ldots\ldots\ldots\ldots \text{area}\ [\mu m^2]$

Equation 1: Calculation of the fiber length based on the rectangle model

$$d = \frac{P}{4} - \sqrt{\left(\frac{P}{4}\right)^2 - A}$$

$l \ldots\ldots\ldots\ldots \text{length}\ [\mu m]$

$p \ldots\ldots\ldots\ldots \text{perimeter}\ [\mu m]$

$A \ldots\ldots\ldots\ldots \text{area}\ [\mu m^2]$

Equation 2: Calculation of the fiber diameter based on the rectangle model

Two limitations must be taken into consideration. The fibers have to have a constant fiber diameter and a smooth surface. Both requirements are fulfilled for manmade fibers.

After calculating the length of all fibers following Equation 1 it is possible to determine the fiber length distribution and to characterize the analyzed fiber collective.

3 Experimental Details

3.1 Fiber Samples

Table 1 gives an overview on the used fiber samples. Bright fibers do not contain any additives and are used for textile applications. Dull fibers contain titanium dioxide to produce a satin finish. Nonwoven fibers also contain titanium dioxide and are used for applications like toiletries. FR fibers are heat protection fibers which are used for fire brigade equipment. Smartcell fibers contain zinc oxide and are used for antibacterial applications.

Table 1: Fiber samples

Manufacturing Process	Fiber titer [dtex]	Starting Length [mm]	Additive
Modal	1.3	39	Bright
Modal	1.3	39	Dull
Modal	1.5	39	Bright 6252 (spin-dyed)
Modal	1.5	39	Bright 6476 (spin-dyed)
Modal	1.5	39	Bright 6730 (spin-dyed)
Modal	1.7	40	Dull FR
Tencel	0.9	34	Bright
Tencel	1.3	38	Nonwoven
Tencel	1.7	6	Silicon
Tencel	1.7	38	Nonwoven
Tencel	1.27	38	Bright
Tencel	2.5	38	Smartcell
Tencel	6.7	60	Smartcell
Viscose	1.3	39	Bright
Viscose	1.3	39	Dull
Viscose	3.3	60	Bright
Viscose	4.2	60	Nonwoven
Viscose	5.0	120	Bright
Viscose	17	100	Dull

3.2 Fiber Grinding

For fiber grinding an Alpine Hosokawa Cutting Mill was used. Figure 1 shows the mill, which has pilot plant scale. Detailed information is listed in Table 2.

Table 2: Detailed information cutting mill

Type	Alpine Hosokawa Ro20/12
Power	4 kW
Airflow	Side Channel Blower 1.5 kW
Rotation Speed	1.500 $Umin^{-1}$
Knives	2 flying / 3 fixed
Rotor	Width 120 mm / Diameter 200 mm

Figure 1: Pilot plant scale cutting mill

Sieves of three different mesh sizes (3 mm, 2 mm and 0.5 mm) and two different shapes (circular mesh and CONIDUR) were used. Figure 2 photographs the two sieve types.

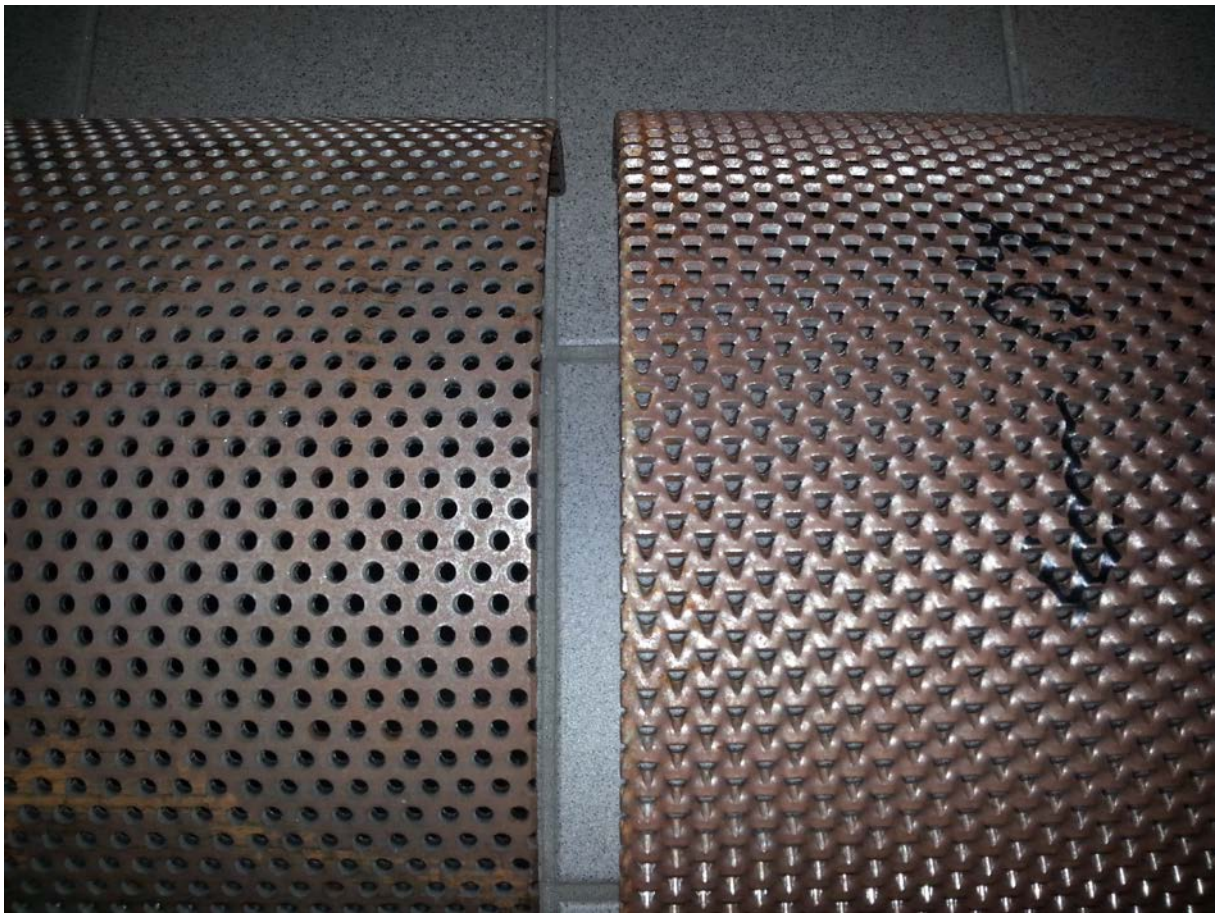

Figure 2: Used sieves; left: circular mesh, right: CONIDUR

The grinding procedure is described in Figure 3.

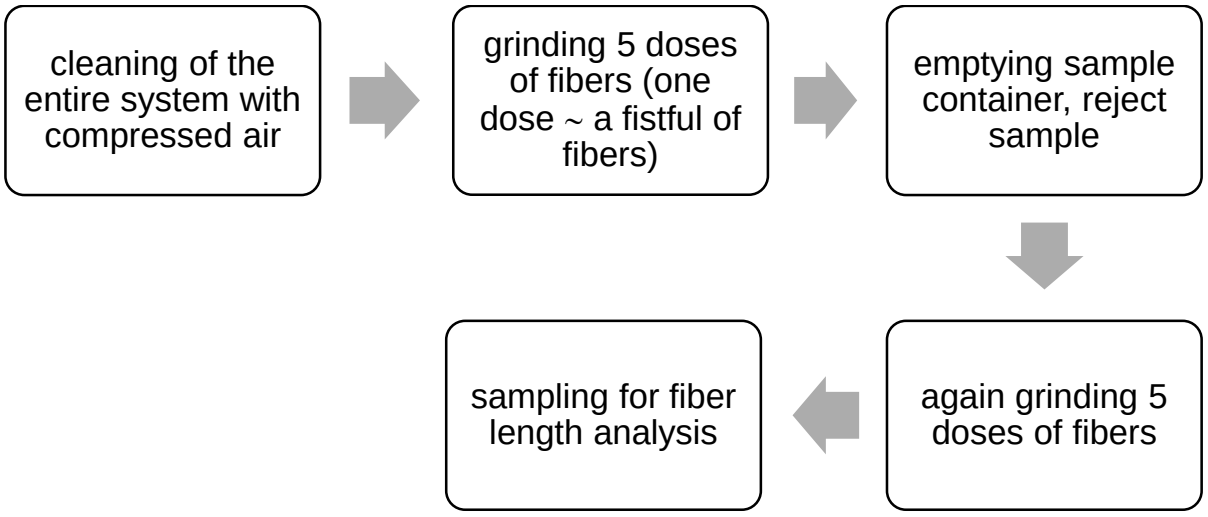

Figure 3: Workflow of grinding experiments

3.3 Fiber Characterization MorFi

The MorFi system was developed by Centre Technique du Papier. It gives reliable and accurate morphological characteristics of fibers, shives and fine elements by recording images of a flowing suspension. The software is using a set of criteria concerning the dimensions and the shape of the particles to separate these three components. For this work only the characterization of fiber length was used. The MorFi System evaluates a particle as a fiber if its length is larger than 100 µm and if its width is larger than 5 µm. The system contains an electromechanical structure, which is connected to a hardware unit. A hydraulic unit and a measurement cell are also part of it [37]. Figure 4 shows the workflow for characterizing the ground fiber samples with MorFi.

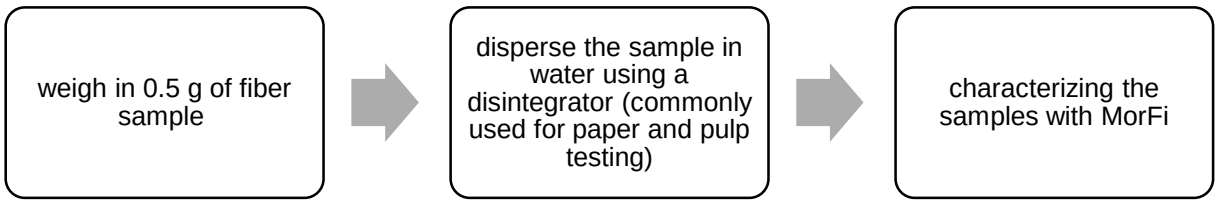

Figure 4: Workflow fiber characterization with MorFi

Grinding and characterization procedures were carried out two times to avoid mistakes based on coincident.

3.4 Fiber Dispersion and Image Recording with Scanner and Microscope

As an alternative to the MorFi system the Olympus BX 61 Microscopy System (resolution 1.38 µm/pixel) and a Photo Scanner, EPSON V500 Photo (resolution 4 µm/pixel), were used for recording images.

For dry dispersion an Occhio Vacuum Disperser was used, for wet dispersion the fibers were either dispersed in water using a disintegrator or in PEG 400 (and other dispersion media for media screening) using a large tumbler and a magnetic stirrer.

Small volume samples were dispersed through shaking in a sample bottle. For image recording the dispersion was put on a petri dish. Approximately 1500 fibers were measured. Figure 5 summarizes the workflow of sample preparation and image recording for the comparative analysis.

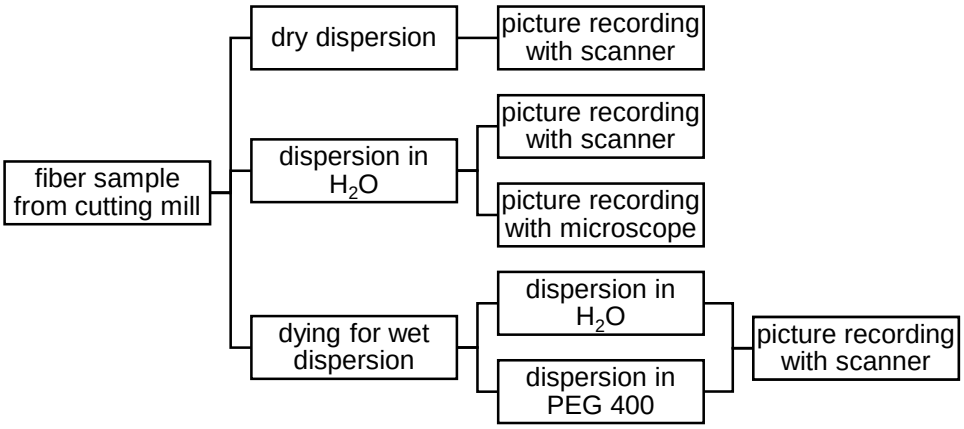

Figure 5: Workflow of image recording using a photo scanner or a microscope

3.5 Fiber Dying

Due to contrast problems it was necessary to dye white fiber samples before analyzing them with the scanner. Colored fibers could be analyzed without dying. For dying the reactive dye Novacron Black NN was used. The dying procedure was based on a scheme found in literature and was slightly amended [38]. A stock solution of distilled water containing 80 g/l sodium chloride, 40 g/l sodium carbonate and 2 g/l sodium hydroxide was prepared. 0.5 g fiber sample were dispersed in 35 ml of this stock solution and about 0.04 g (one spattle top) of Novacron Black NN were added. This mixture was then stirred using a magnetic stirrer and heated following the temperature profile shown in Figure 6.

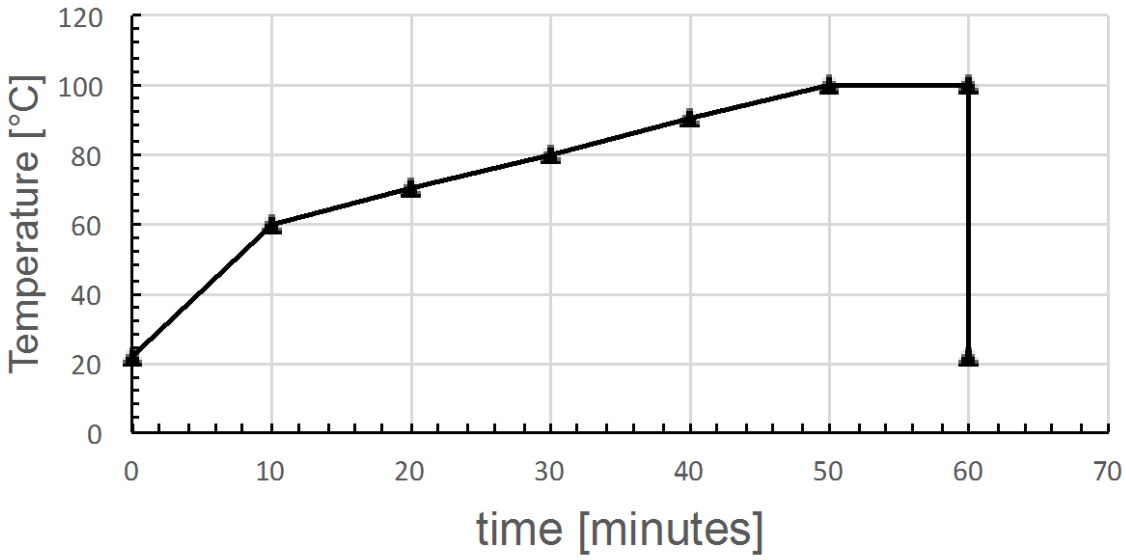

Figure 6: Temperature profile for fiber dying reaction

After the dying reaction the fiber/dying solution mixture was diluted with distilled water and again stirred for washing. Afterwards the fibers were separated using a blue ribbon filter, a strainer, a feeding bottle and a water-pump-jet. After separation the fibers were dried by sucking air through the formed fiber fleece on the filter. Afterwards the fiber sample was ready for characterization.

3.6 Data Processing

3.6.1 MorFi Software

For characterizing fibers a standard operation procedure (SOP) was created. 10.000 fibers were analyzed concerning their length and divided into hundred size classes of same size from 100 µm to 5000 µm. For every class the MorFi software computed the amount of length (in percent). The data from the MorFi system have been exported to an Excel sheet and based on these values the length distribution was calculated using Equation 3.

$$q_{1,i} = \frac{l_i}{\Delta x_i}$$

$q_{1,i}$ … … … … fiber length density function [μm^{-1}]
l_i … … … … portion of length of class i [−]
Δx_i … … … … class width of class i [µm]

Equation 3: Calculation of fiber length density function q_1

The sum function was calculated following Equation 4.

$$Q_1(x_*) = \int_{x_{min}}^{x*} q_1(x)dx$$

Q_1 … … … … fiber length sum function [−]
x_{min} … … … … shortest fiber length [µm]
x_* … … … … control variable describing the fiber length [µm]

Equation 4: Calculation of fiber length sum function Q_1

The average fiber length was calculated using a polynomial interpolation of sixth degree of the sum function. This interpolation was solved for $Q_1 = 0.5$. This so-called median value was used as an average fiber length to characterize the fiber samples.

3.6.2 AnalySIS Software and Scientific Counter Software

AnalySIS is the software, which is provided with the Olympus Microscopy System. With this software it is possible to analyze images, which have been recorded with the microscopy system or with other image recording systems. AnalySIS can measure a series of established particle parameters. In this case area and perimeter of the fiber samples were measured. AnalySIS is required by the microscopy system hardware for operating.

Scientific Counter is a self-sustaining software package for image analysis of images recorded with microscopes or other image recording systems. It is provided from the German company DatInf. Like AnalySIS it can measure a series of particle parameters. Again area and perimeter of the fiber samples were measured.

Based on the fiber data measured with AnalySIS or Scientific Counter the fiber length was computed using the rectangle model of Beck [36]. Analogue to the procedure of

the MorFi the analyzed fibers were divided into 100 classes from 100 µm up to 5000 µm. Afterwards the portion of length of every class of the collective was calculated. Based on this data the fiber length density and sum functions were calculated using again Equation 3 and Equation 4. The average fiber length was calculated as described in chapter 3.6.1.

3.7 Determining Bulk Density

For the determination of the bulk density a beaker with a defined volume of 226 ml was used (the volume was determined via volumetric measurement). Directly after grinding a sample was taken and filled into the beaker, which was tared before. The overlaying fibers were stripped using a ruler and the beaker with the sample was weighed. Based on the weight of the sample and the known volume of the beaker it was possible to determine the bulk density in grams per liter. This procedure was carried out three times and the average value was calculated.

4 Results

In the following chapters the results of this work are presented and summarized.

4.1 Grinding Process

This chapter deals with the results of the grinding experiments carried out to get more information about the grinding process.

4.1.1 Influence of different Fiber Parameters

Figure 7 shows the results for the experiments carried out with Modal fibers. The fibers had a fiber titer of 1.3 dtex and a starting length of 39 millimeters. The left diagrams show the density functions and the right ones the sum functions. Based on the sum functions an average fiber length of 809 µm has been calculated.

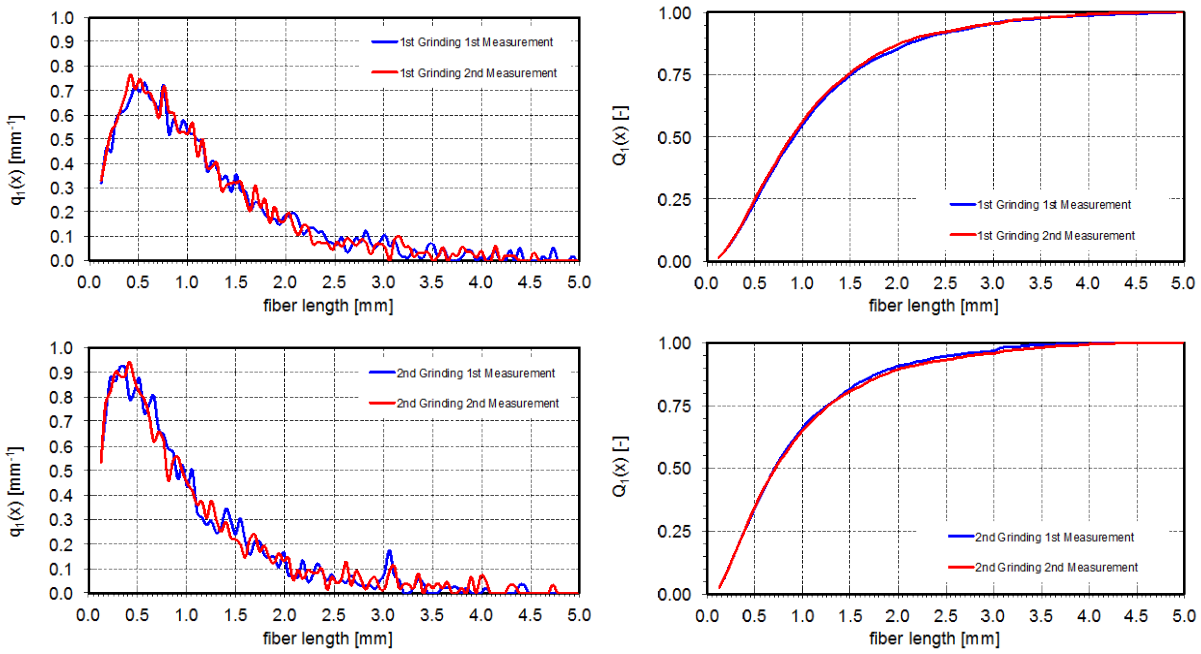

Figure 7: Modal 1.3 39 Bright

Figure 8 summarizes the results for the experiments with Dull Modal fibers. The fibers also had a fiber titer of 1.3 dtex and a starting length of 39 mm but they contained TiO_2 particles for creating a satin finish. An average fiber length of 906 µm has been ascertained.

The fibers used in the experimental series presented in Figure 9 contained pigments (pigment number 6252). They showed a fiber titer of 1.5 dtex and a starting length of 39 mm. Based on the sum functions an average fiber length of 724 µm was calculated.

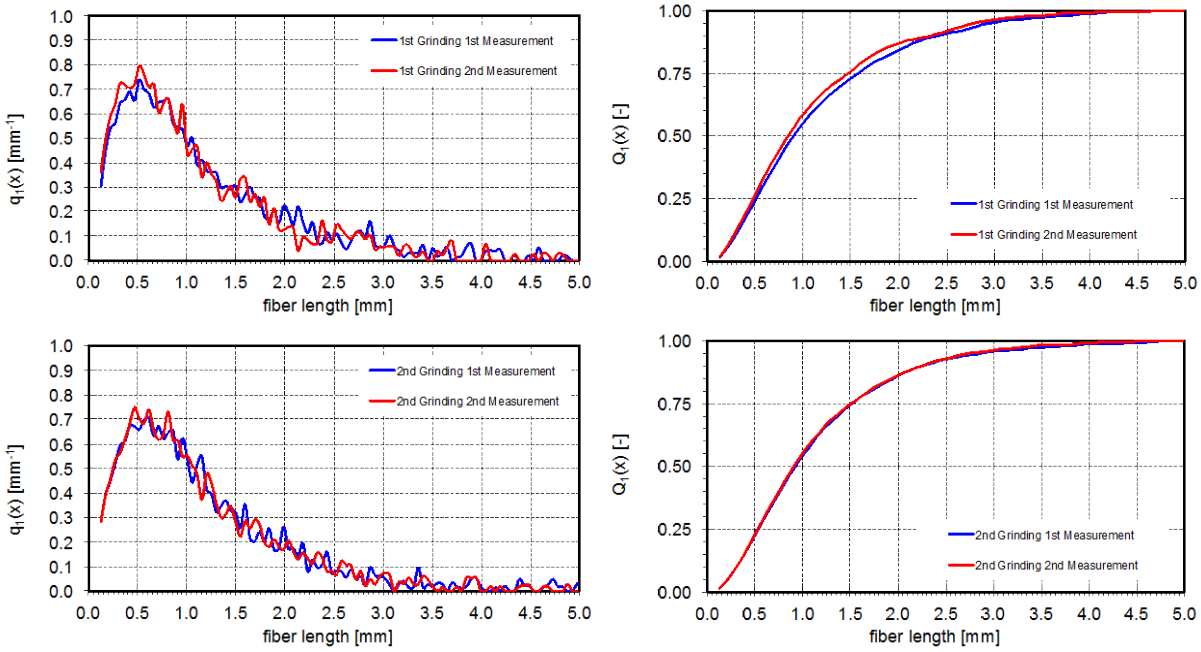

Figure 8: Modal 1.3 39 Dull

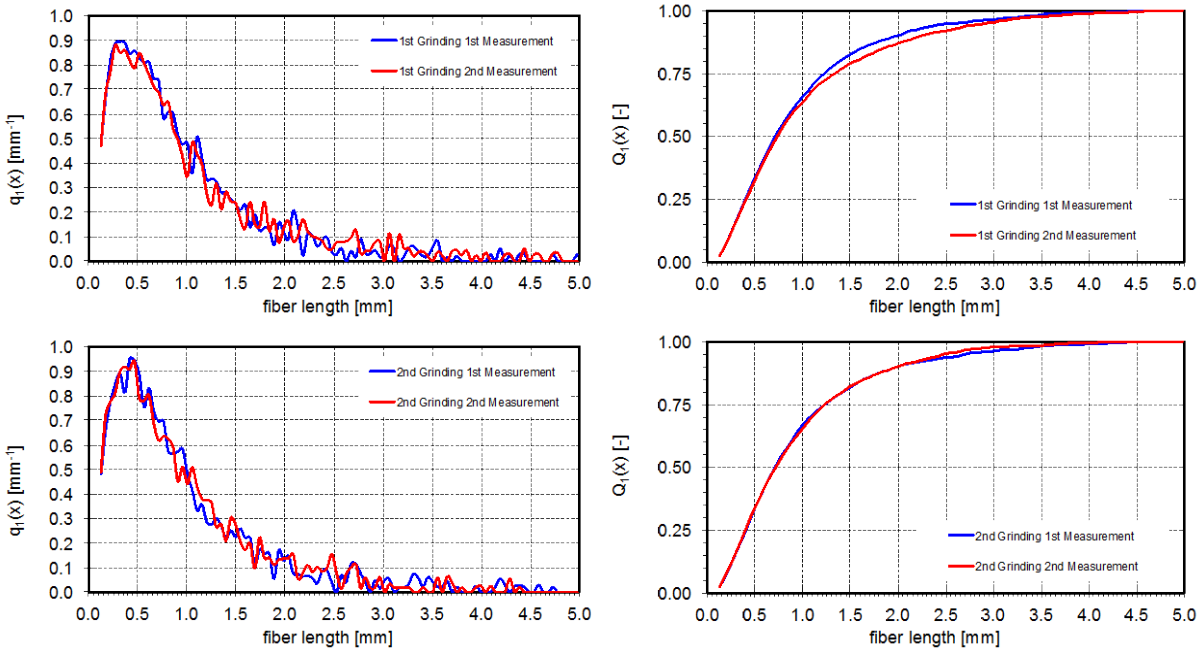

Figure 9: Modal 1.5 39 Bright 6252

Another experimental series was carried out with fibers containing the color pigment 6476. These fibers had the same fiber parameters like Modal fibers with pigment 6252, only the pigment differed. Figure 10 shows the results. An average fiber length of 839 µm has been calculated.

The fiber sample for the series plotted in Figure 11 contained the pigment 6730. The other fiber parameters were the same like the fibers used for the experiments in Figure 9 and Figure 10 had. This fiber type showed and average fiber length of 737 µm.

The diagrams in Figure 12 show the results for grinding Modal FR fibers. These heat protection fibers had a fiber titer of 1.7 dtex and a starting length of 40 mm. They also contained TiO_2 for producing a satin finish. The analysis gave an average fiber length of 792 µm.

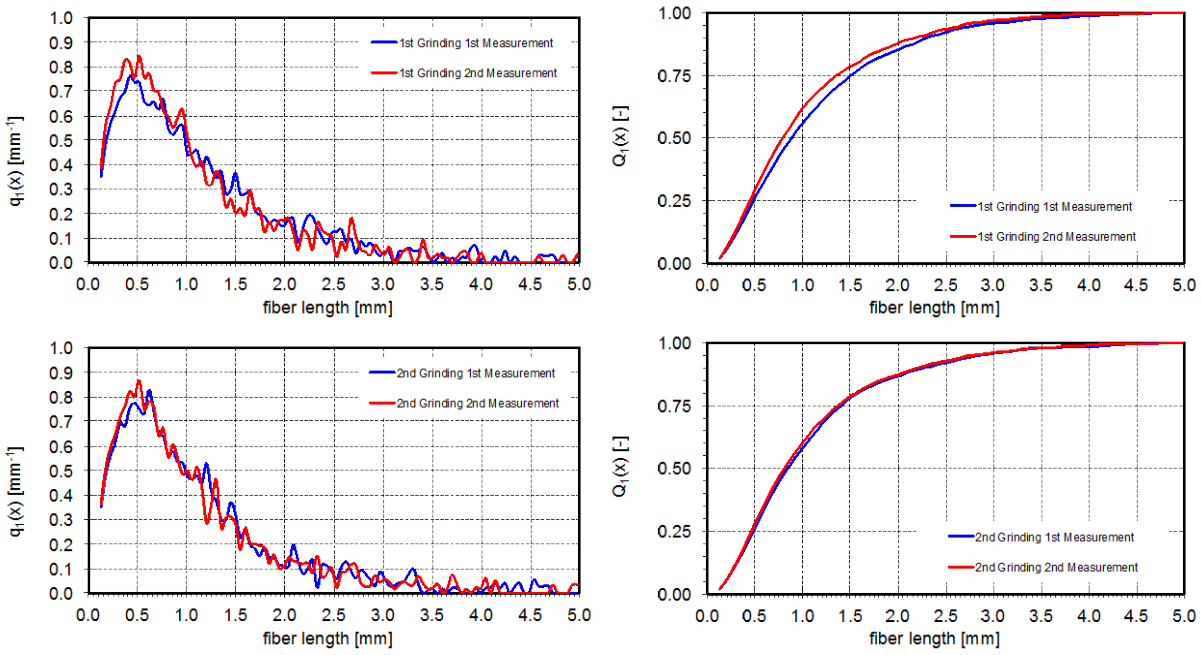

Figure 10: Modal 1.5 39 Bright 6476

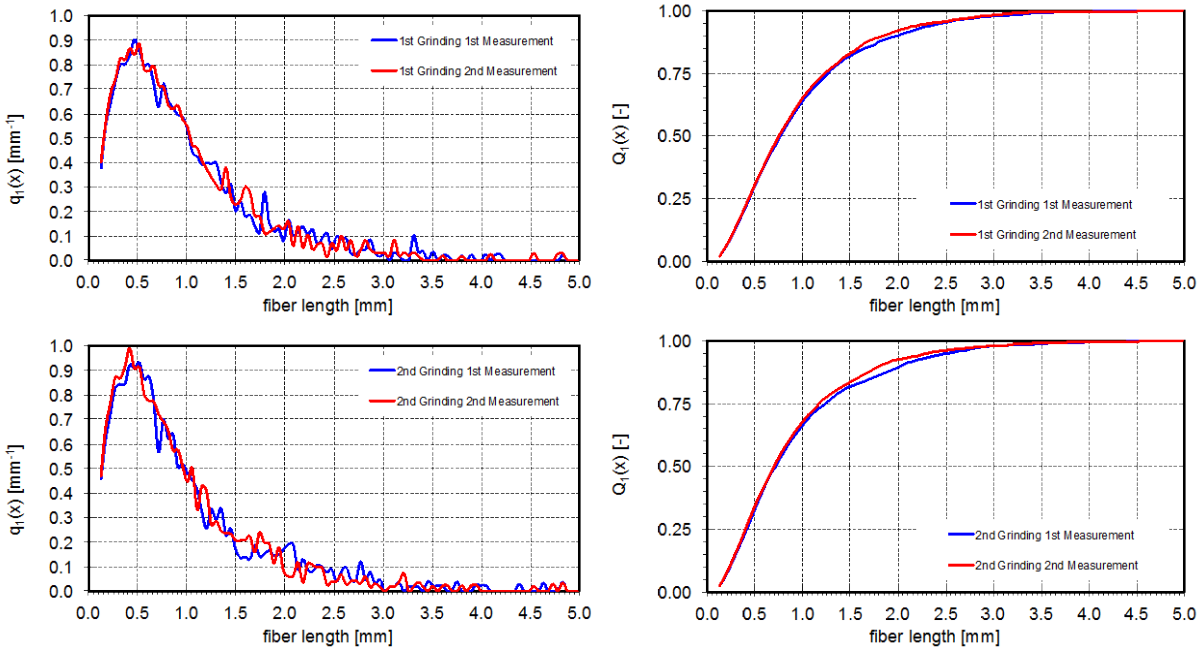

Figure 11: Modal 1.5 39 Bright 6730

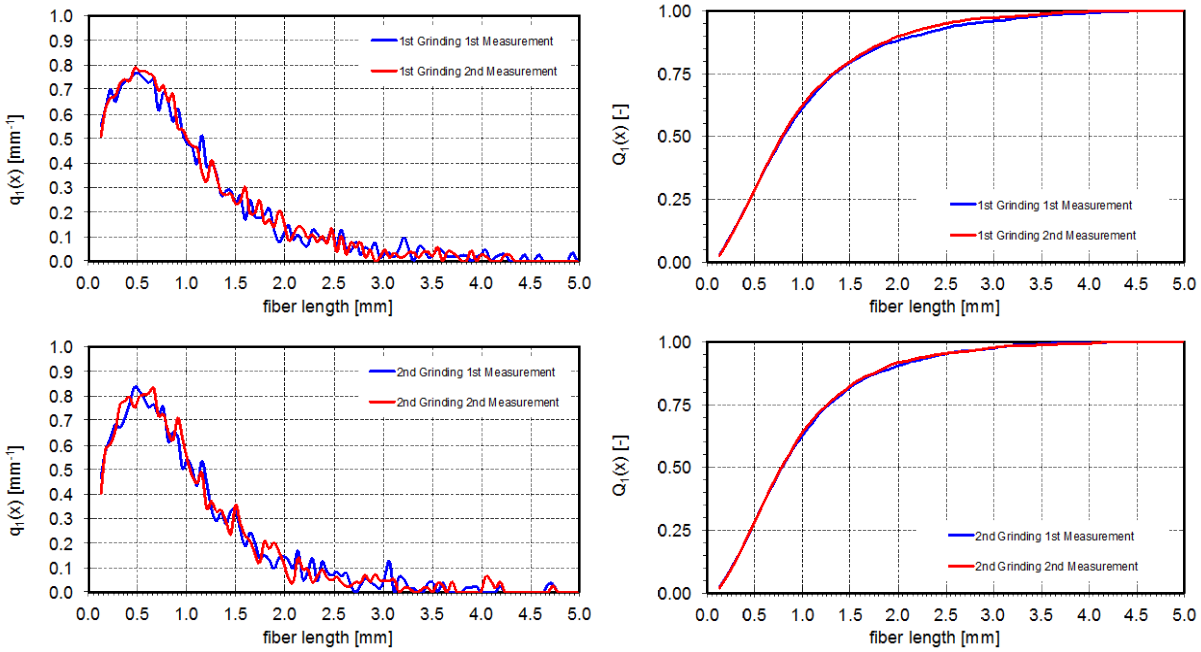

Figure 12: Modal FR 1.7 40 Dull

Tencel fibers also have been investigated concerning their behavior in a grinding process. Figure 13 shows the results for Tencel fibers with a fiber titer of 0.9 dtex and a starting length of 34 mm. The fiber samples did not contain any sizing. An average fiber length of 729 µm has been ascertained.

Also fibers for Nonwoven applications have been investigated. Figure 14 shows the results for analyzing a Tencel fiber sample with a fiber titer of 1.3 dtex and a starting length of 38 mm. Analysis have resulted in an average fiber length of 795 µm.

Figure 15 shows the results for grinding a Tencel sample with 1.7 dtex fiber titer, 6 mm starting length and silicon sizing. An average fiber length of 697 µm was calculated.

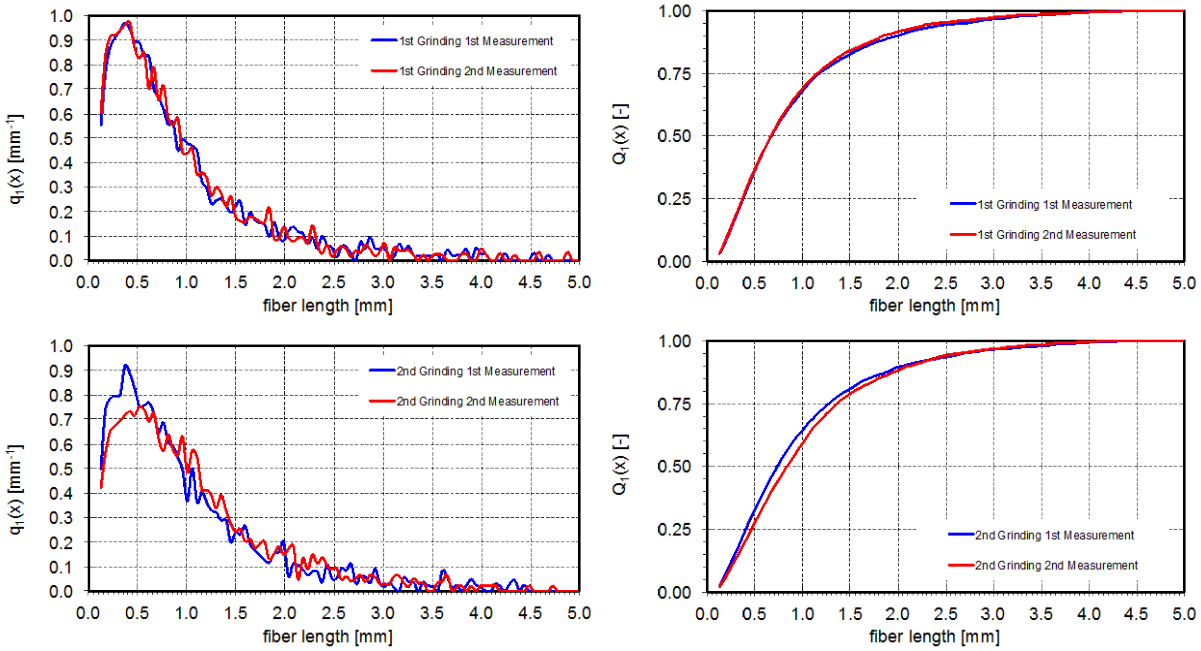

Figure 13: Tencel 0.9 34 Bright

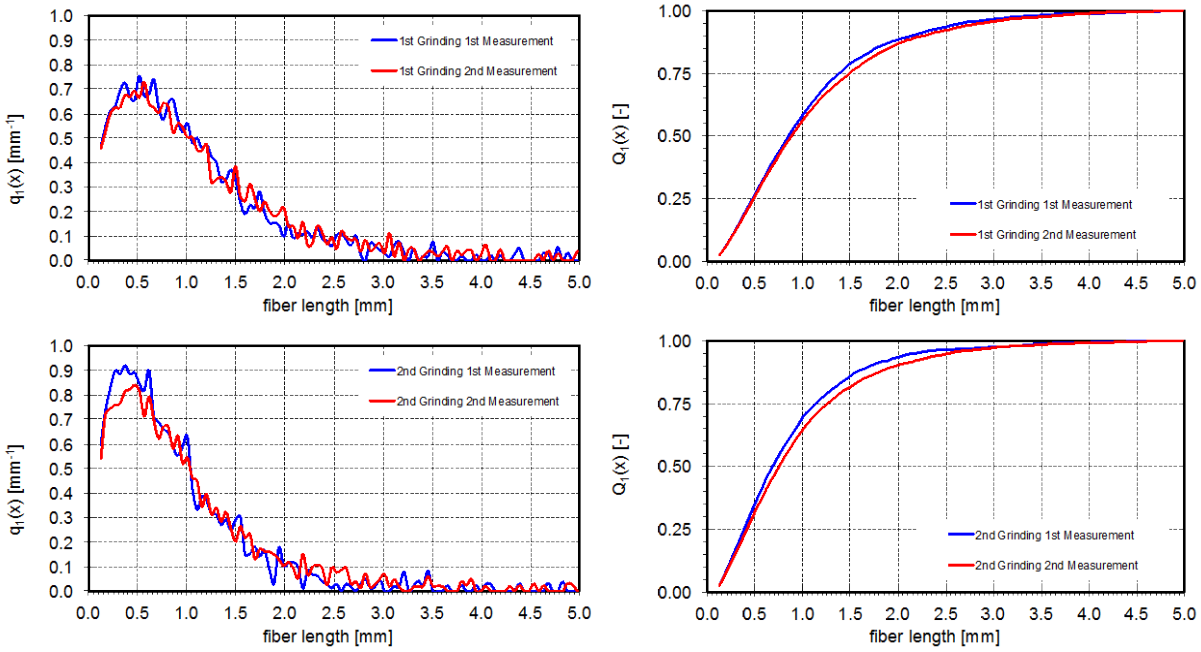

Figure 14: Tencel 1.3 38 Nonwoven

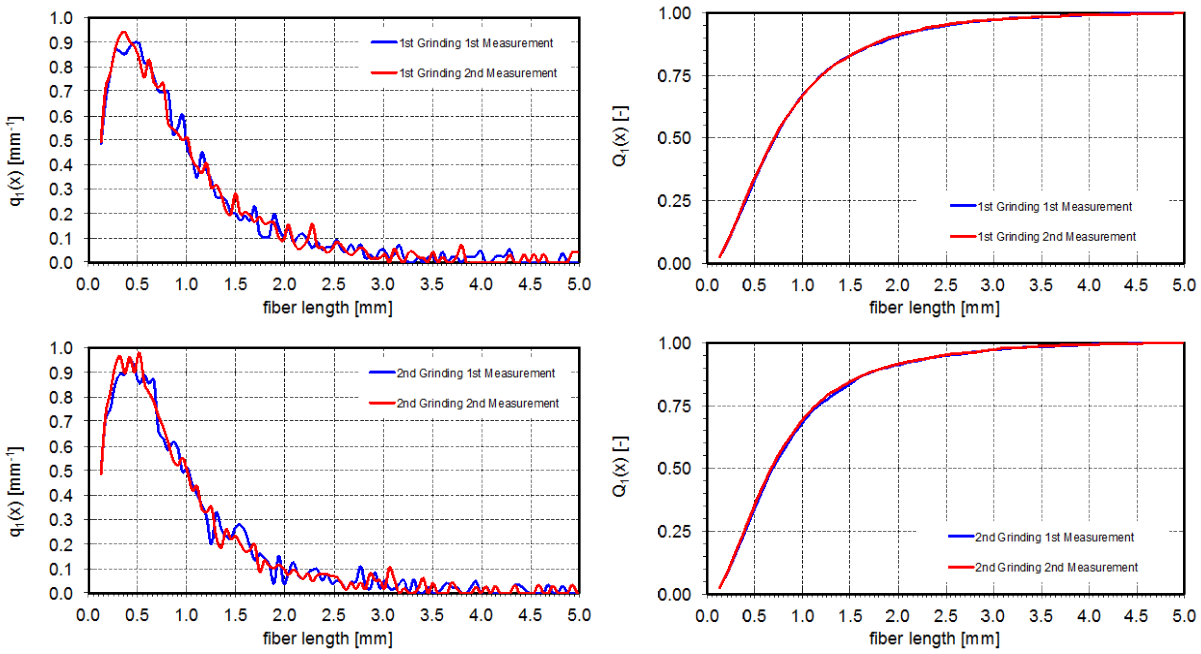

Figure 15: Tencel 1.7 6 Silicon

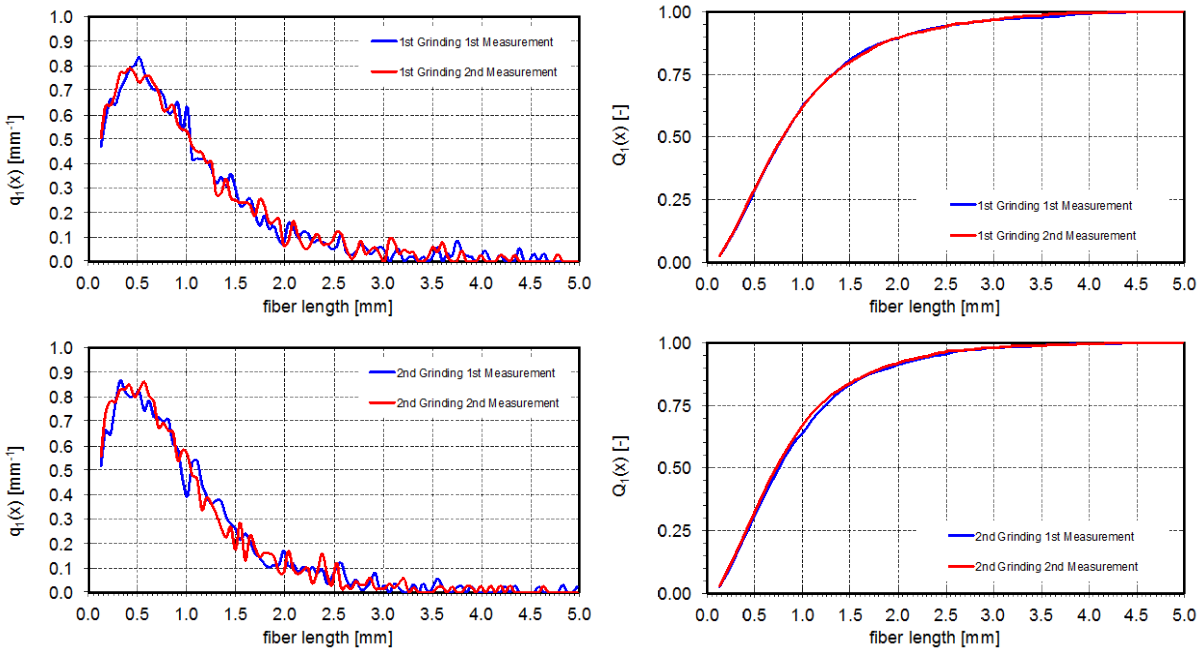

Figure 16: Tencel 1.7 38 Nonwoven

A second Tencel Nonwoven sample has been analyzed with the same starting length but with different fiber titer. The sample had a fiber titer of 1.7 dtex. It showed an average fiber length of 767 µm. Figure 16 presents the results.

Also Bright Tencel samples for textile usage have been investigated. A sample with 1.27 dtex fiber titer and 38 mm starting length has been analyzed. An average fiber length of 845 µm was determined. The results are plotted in Figure 17.

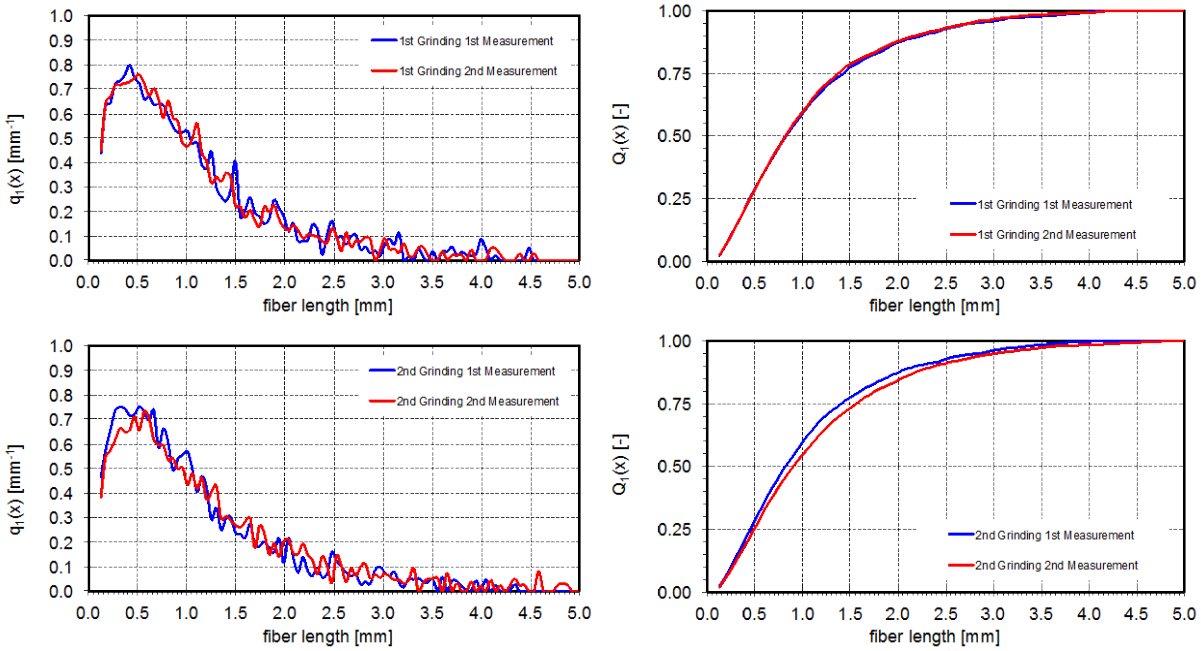

Figure 17: Tencel 1.27 38 Bright

Tencel fibers are also sold equipped with ZnO for antibacterial applications. Two types of such Smartcell fibers have been analyzed.

The results for the first sample with 2.5 dtex fiber titer and 38 mm starting length are summarized in Figure 18. An average fiber length of 902 µm was determined.

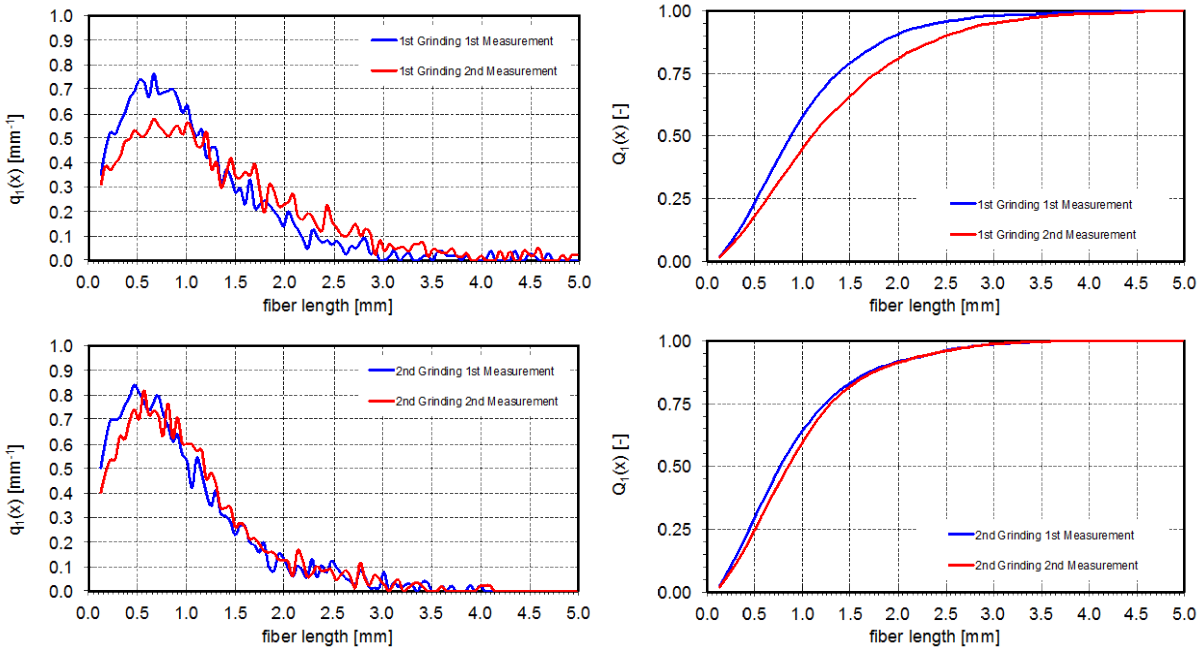

Figure 18: Tencel 2.5 38 Smartcell

The results for the second sample with 6.7 dtex fiber titer and 60 mm starting length are summarized in Figure 19. Analysis gave an average fiber length of 1173 µm.

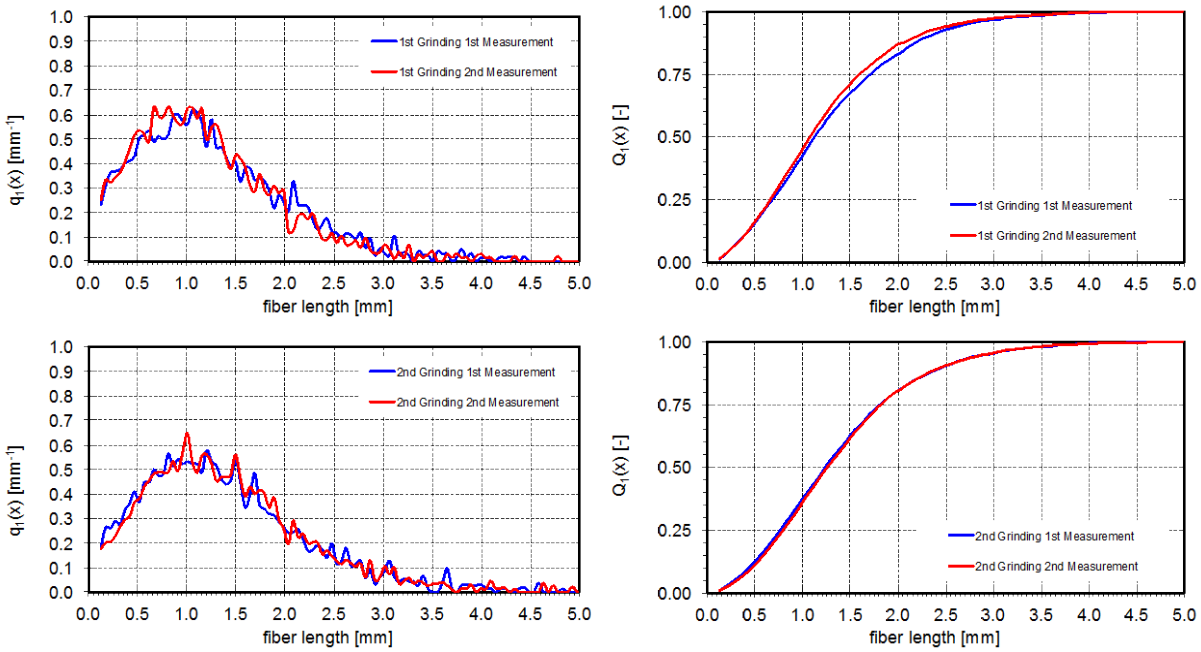

Figure 19: Tencel 6.7 60 Smartcell

Viscose fiber samples have also been analyzed. Figure 20 shows the results for a Viscose fiber sample with 1.3 dtex fiber titer and a starting length of 39 mm. Based on the sum functions an average fiber length of 804 µm was determined.

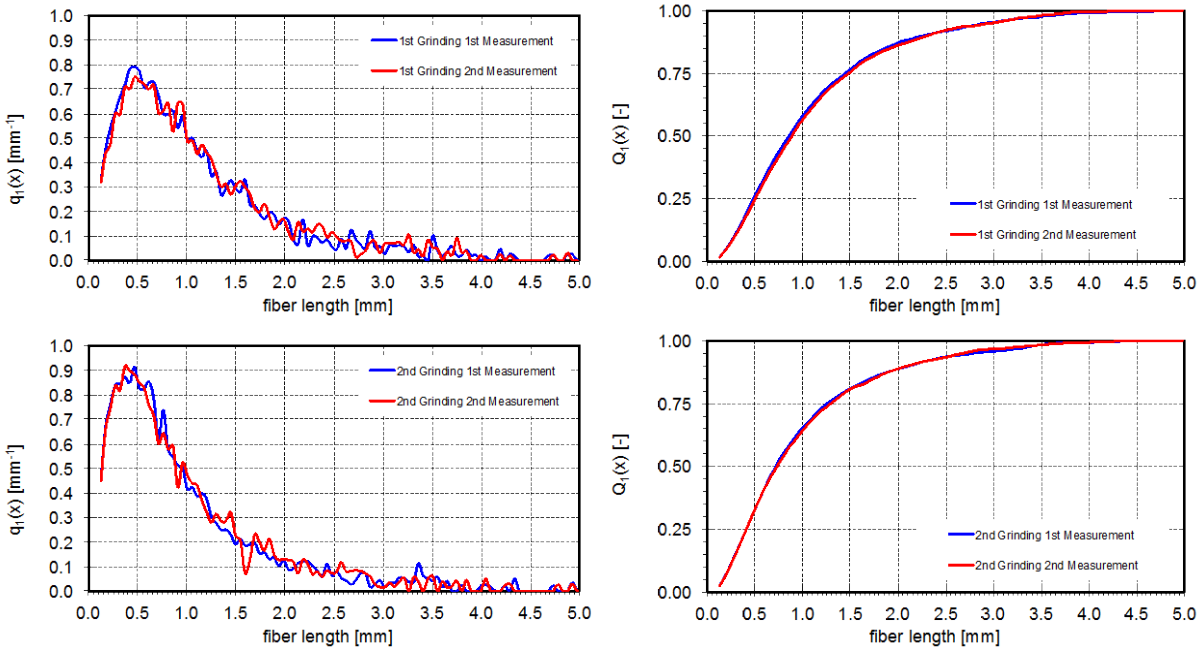

Figure 20: Viscose 1.3 39 Bright

There were also experiments carried out with Dull Viscose fibers, which had the same fiber parameters. Figure 21 shows the results. An average fiber length of 814 µm was calculated.

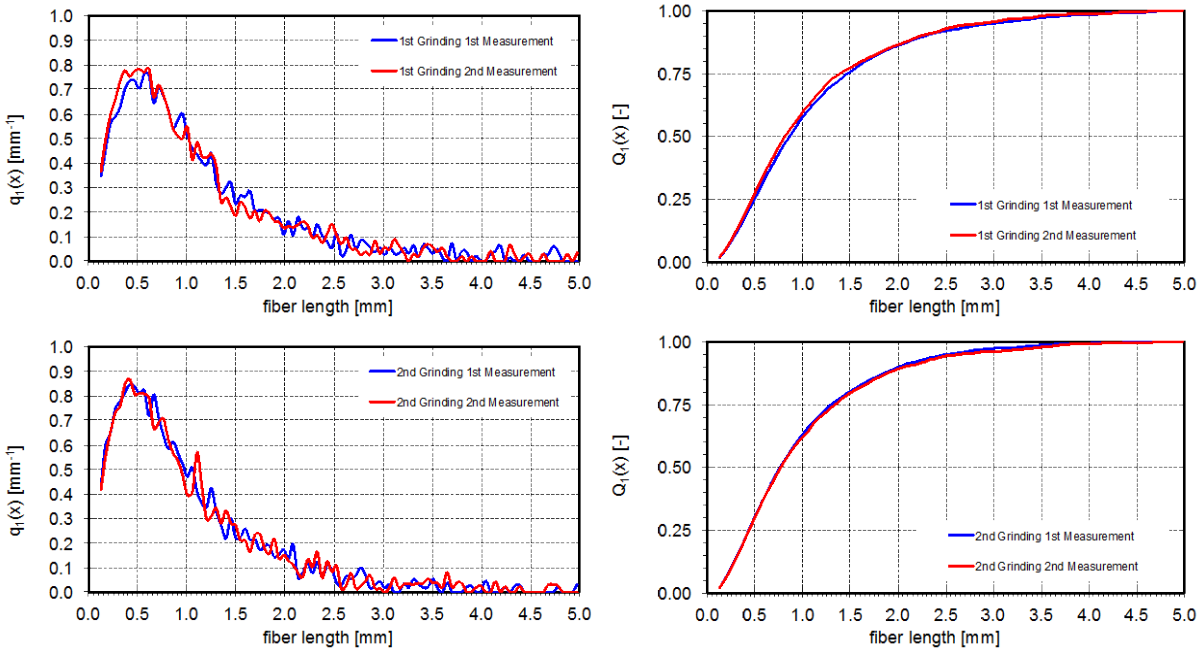

Figure 21: Viscose 1.3 39 Dull

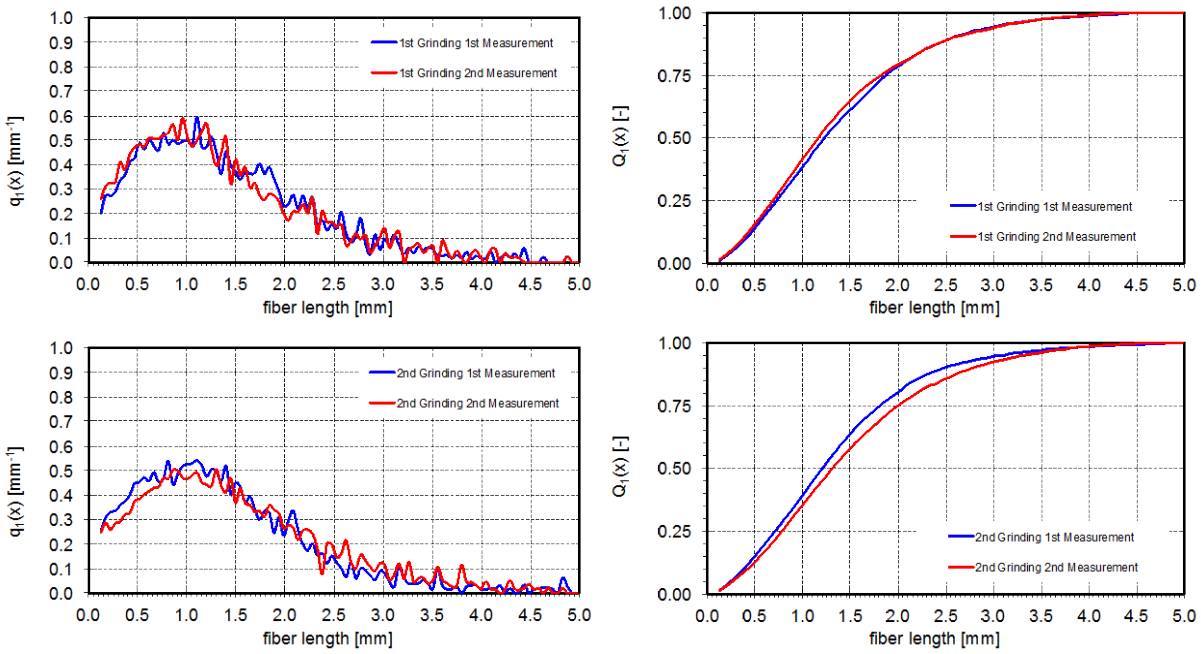

Figure 22: Viscose 3.3 60 Bright

To get more information about the influence of the fiber titer and starting length on the resulting fiber length, Viscose fiber samples with different fiber titers and starting lengths have been ground and analyzed. Figure 22 shows the results for a Viscose fiber sample with 3.3 dtex fiber titer and a starting length of 60 mm. An average fiber length of 1226 µm was measured.

A Dull Viscose sample with 4.2 dtex fiber titer and 60 mm starting length gave the following results showed in Figure 23. An average fiber length of 1142 µm was measured.

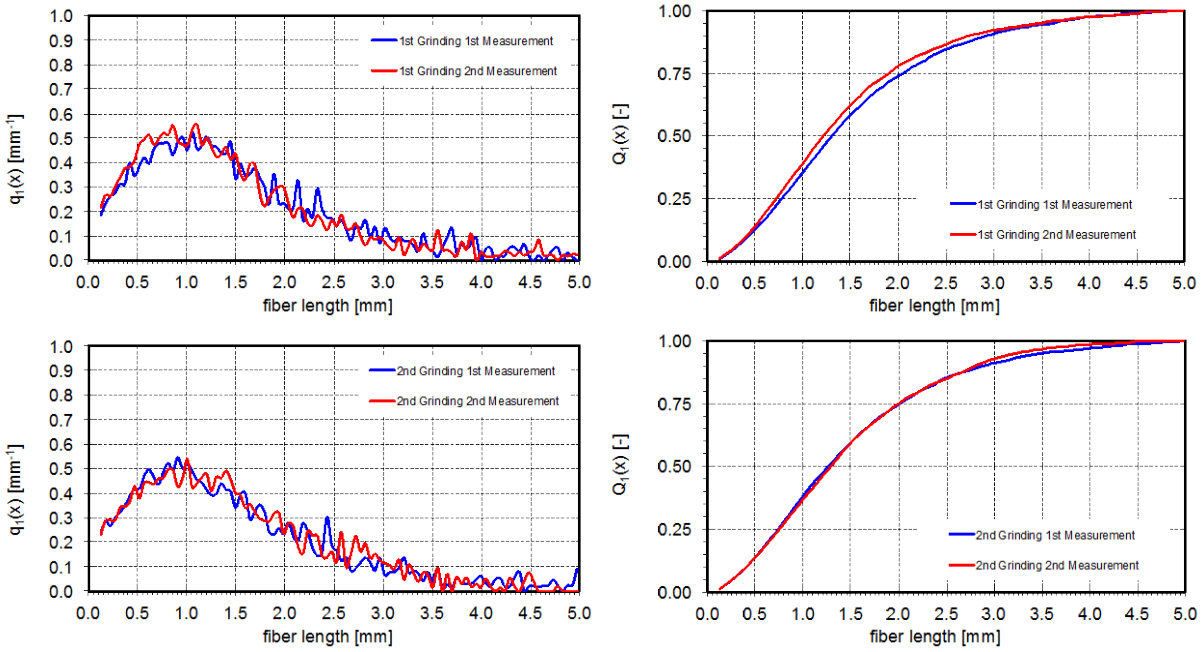

Figure 23: Viscose 4.2 60 Dull Nonwoven

Figure 24 shows the results for grinding a Viscose fiber sample with 5.0 dtex titer and 120 mm starting length. An average fiber length of 1288 µm was determined.

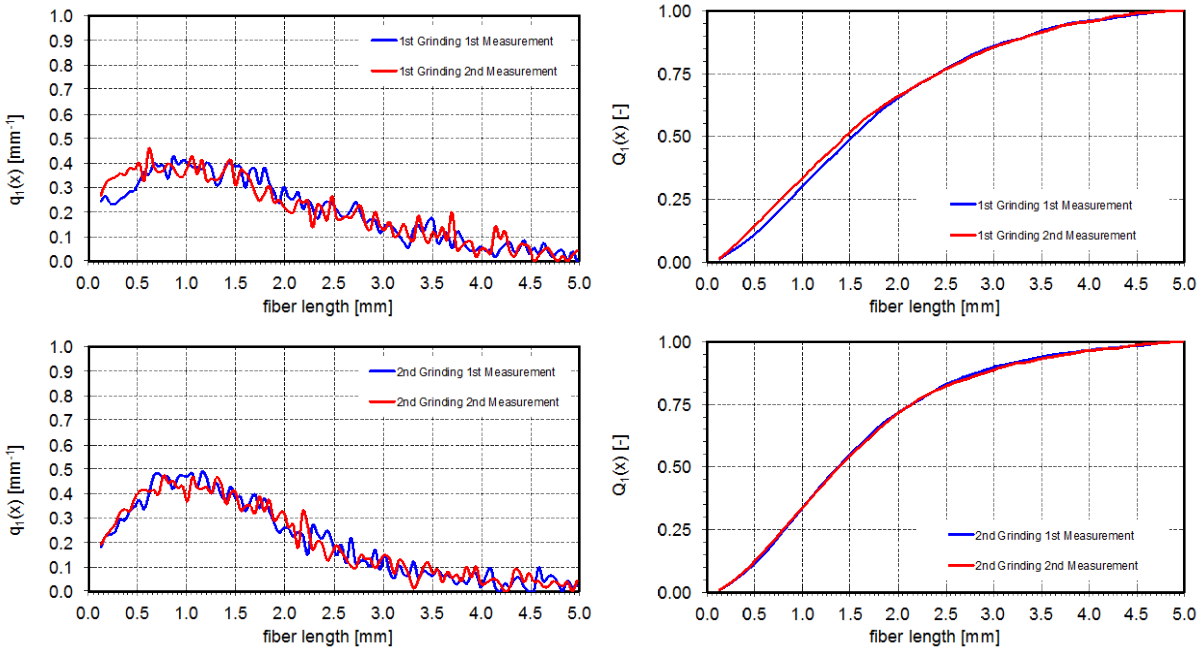

Figure 24: Viscose 5.0 120 Bright

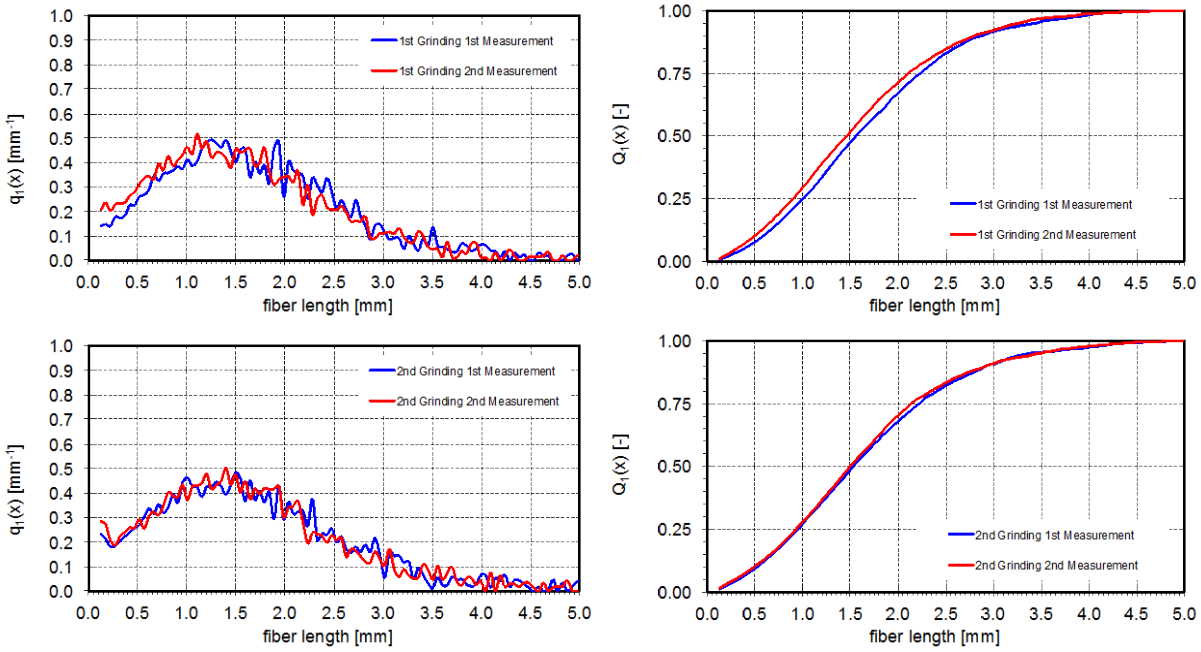

Figure 25: Viscose 17 100 Dull

The last analyzed samples were Dull Viscose fibers, which had a fiber titer of 17 dtex and a starting length of 100 mm. Based on Figure 25 an average fiber length of 1512 µm was determined.

Table 3 and Figure 26 summarize all the results obtained from MorFi.

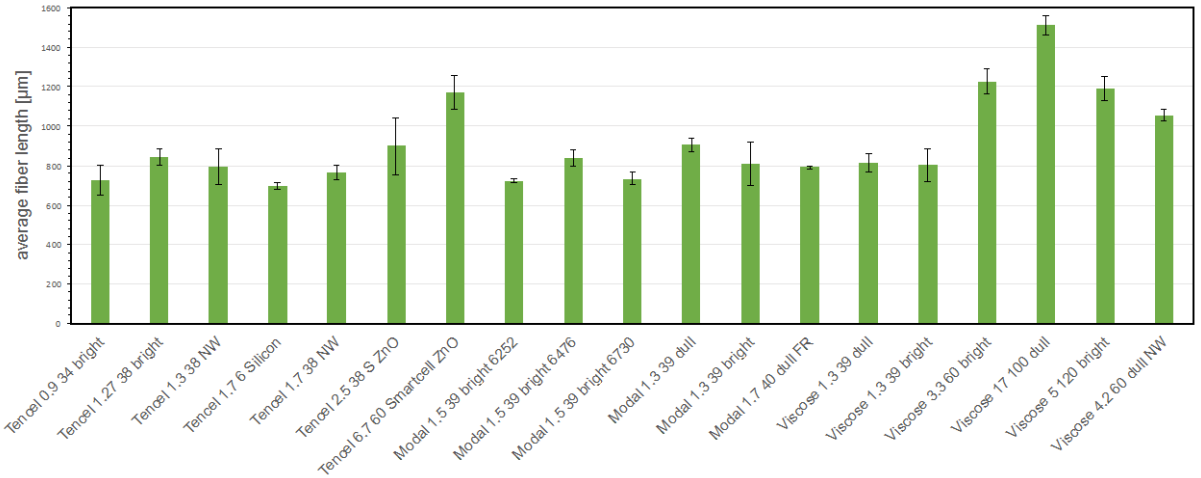

Figure 26: Summary of grinding experiments

Table 3: Summarized results grinding experiments

Fiber sample	Average fiber length [µm]
Modal 1.3 39 Bright	809
Modal 1.3 39 Dull	861
Modal 1.5 39 Bright 6252	724
Modal 1.5 39 Bright 6476	839
Modal 1.5 39 Bright 6730	737
Modal FR 1.7 40 Dull	792
Tencel 0.9 34 Bright	729
Tencel 1.3 38 Nonwoven	795
Tencel 1.7 6 Silicon	697
Tencel 1.7 38 Nonwoven	767
Tencel 1.27 38 Bright	845
Tencel 2.5 38 Smartcell	902
Tencel 6.7 60 Smartcell	1173
Viscose 1.3 39 Bright	804
Viscose 1.3 39 Dull	814
Viscose 3.3 60 Bright	1226
Viscose 4.2 60 Dull Nonwoven	1142
Viscose 5.0 120 Bright	1288
Viscose 17 100 Dull	1512

4.1.2 Influence of different Process Parameters – Sieves

In this part of the work, the influence of the sieve on the resulting fiber length was investigated. Figure 27 and Figure 28 show the results for the grinding experiments. Two different sieve types (circular mesh and CONIDUR) and three different mesh sizes were used in these experiments.

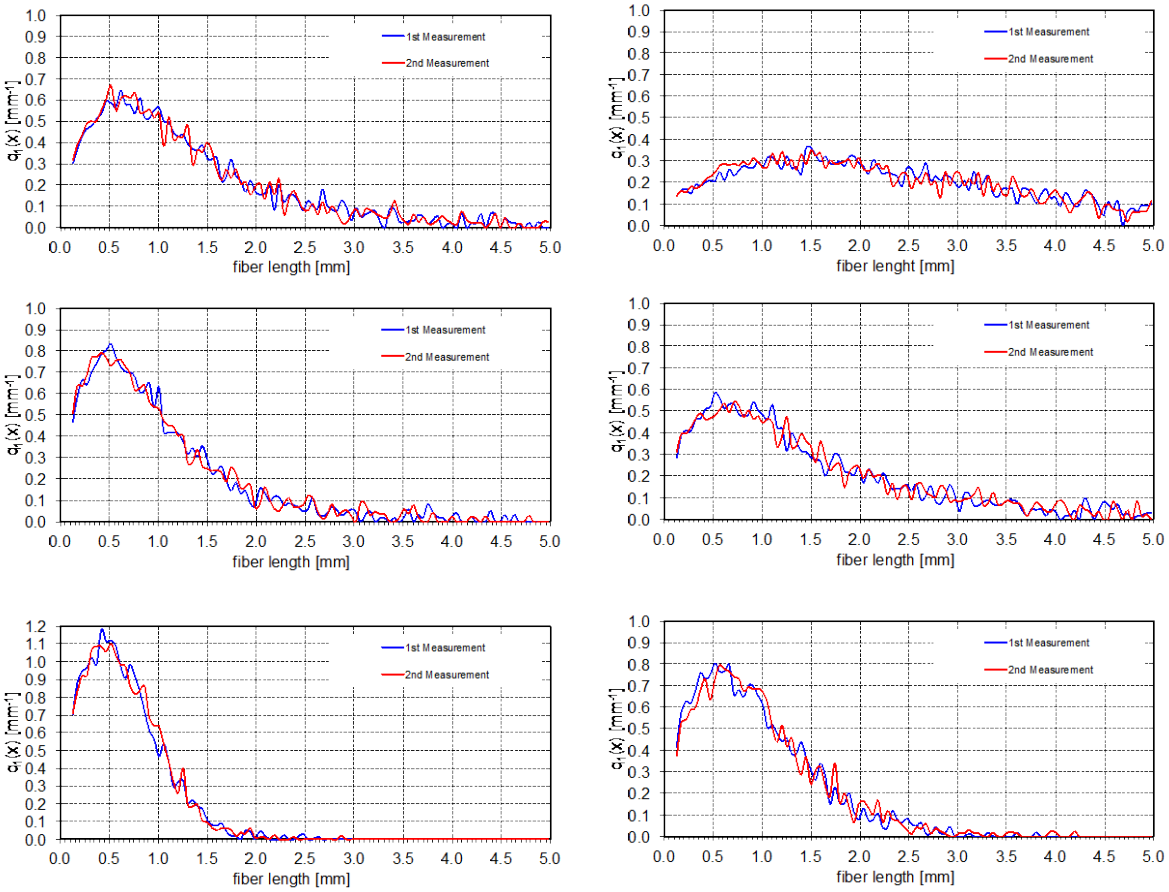

Figure 27: density functions for circular mesh (left) and CONIDUR (right) sieves with 3 mm, 2 mm and 0.5 mm mesh size (top down)

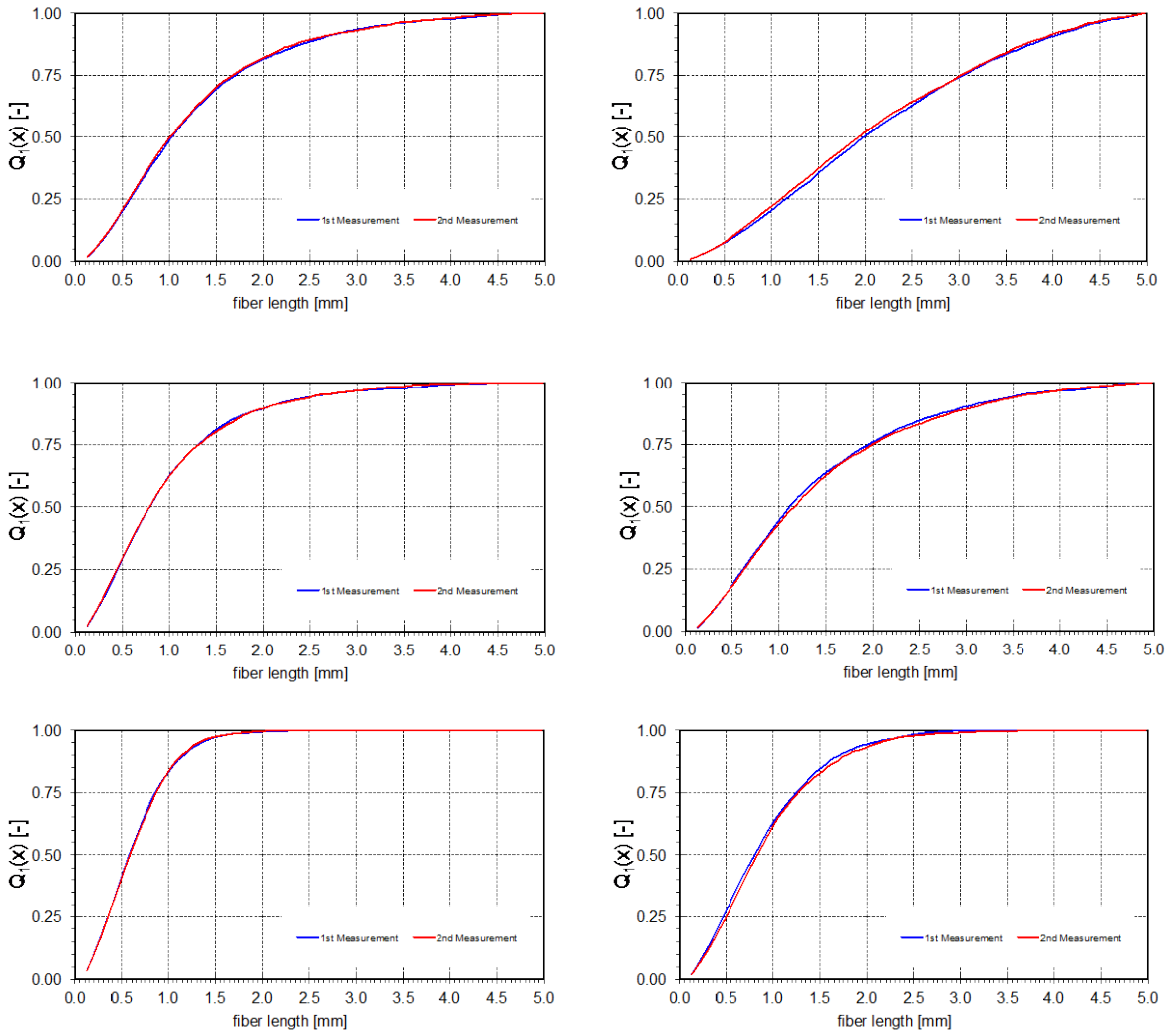

Figure 28: sum functions for circular mesh (left) and CONIDUR (right) sieves with 3 mm, 2 mm and 0.5 mm mesh size (top down)

4.1.3 Influence of different Process Parameters – Process Mode

Further experimental series were carried out investigating different process modes. In this series two different kinds of a multi-stage process were analyzed. In the first one fiber samples were ground four times in a row using always a 2 mm sieve of circular mesh. In the second one, fibers were ground three times in a row using sieves from 3 mm circular mesh to 0.5 mm circular mesh. The results are summarized in Figure 29 and Figure 30.

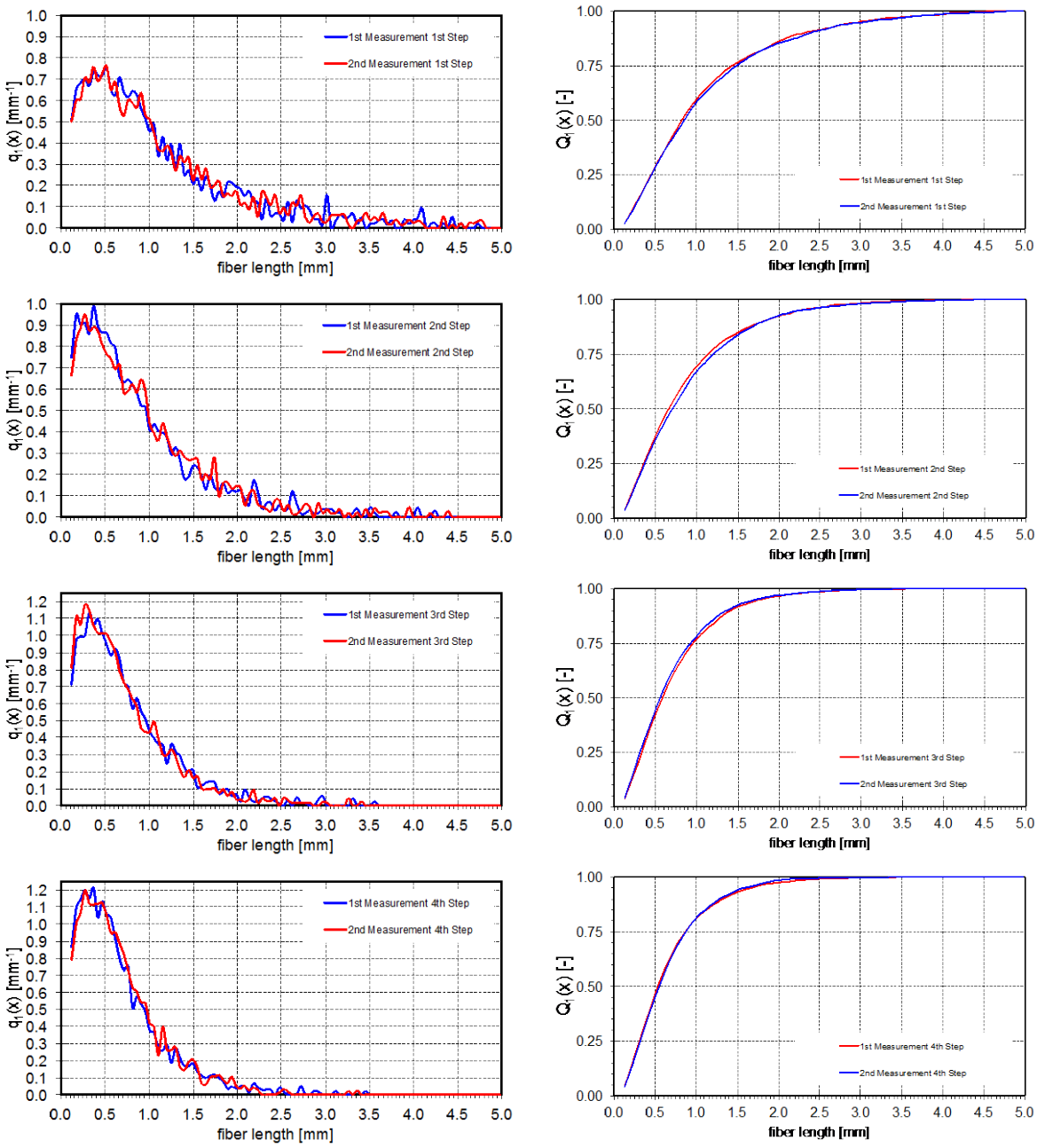

Figure 29: Results for a multi-stage grinding process using 4 times in a row a 2 mm sieve of circular mesh

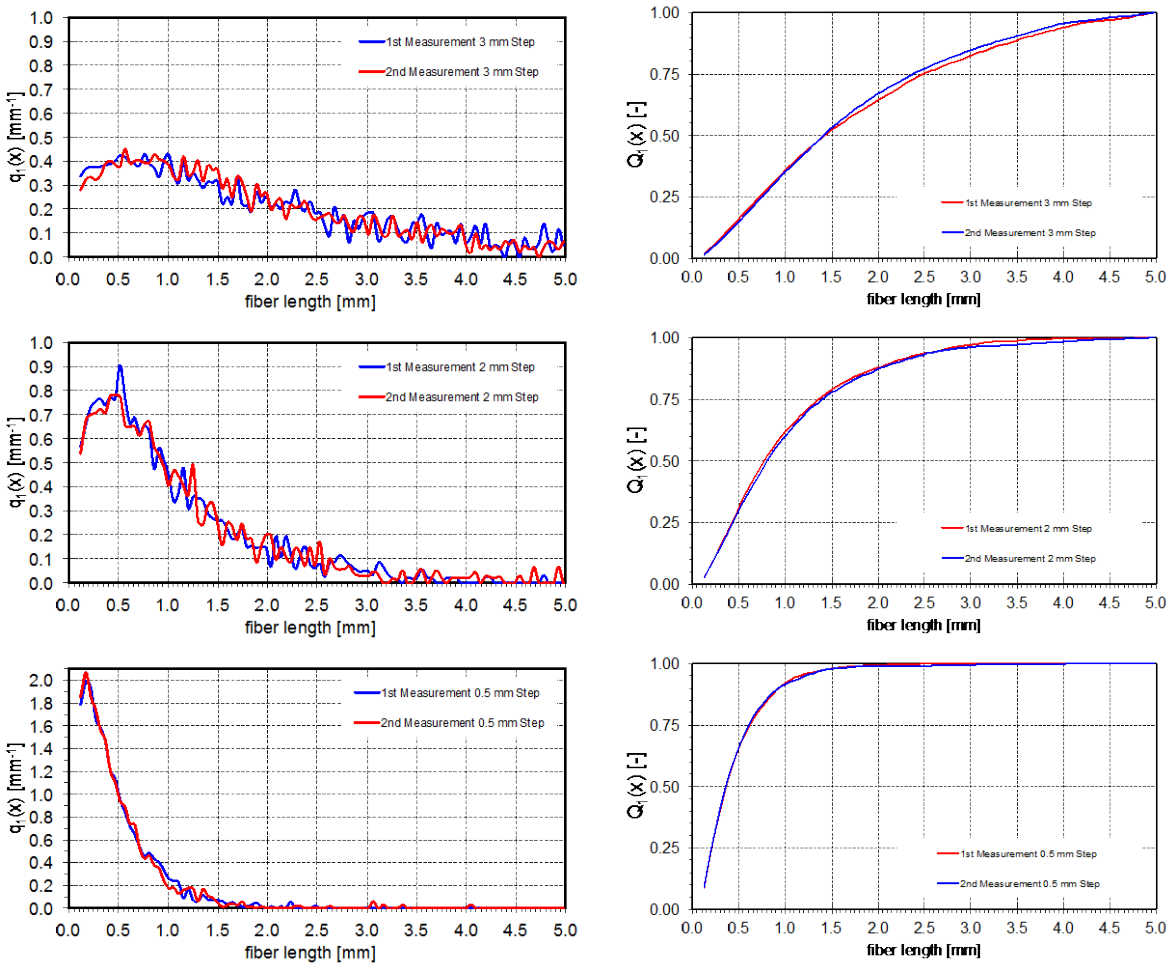

Figure 30: Results for a multi-stage grinding process using sieves of circular mesh from 3 mm to 0.5 mm mesh size

4.1.4 Influence of different Process Parameters – Airflow

The influence of the airflow, which is used to carry the ground fibers out of the cutting mill was also investigated. To get a first insight in how the airflow influences the resulting fiber length, grinding experiments with two different airflows were performed. Figure 31 and Figure 32 show the results for the experiments carried out at 50% and 75% airflow.

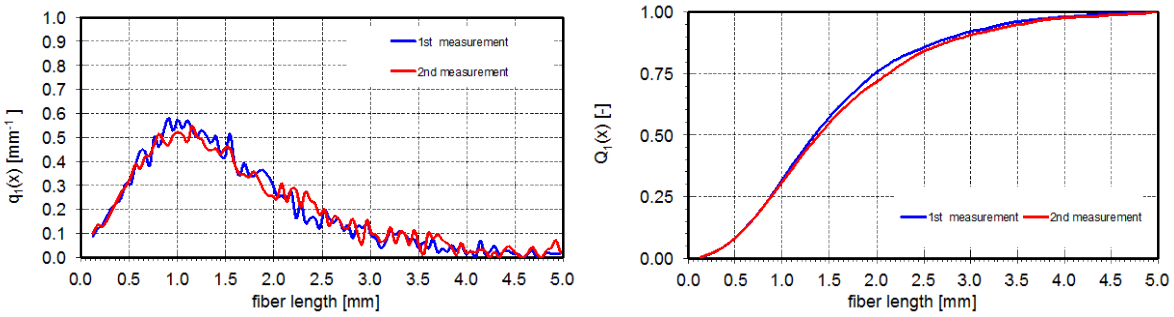

Figure 31: 50% airflow

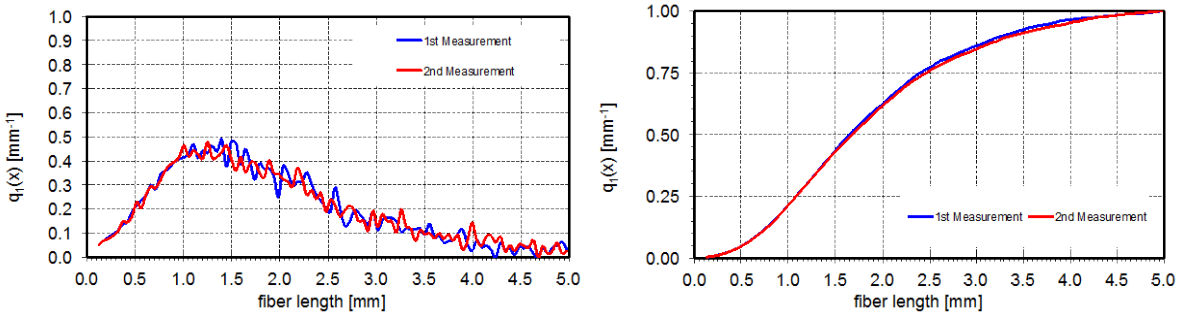

Figure 32: 75% airflow

Table 4 summarizes the results.

Table 4: Summarized results airflow experiments

Airflow [%]	Average fiber length [µm]
50	1367
75	1668

4.2 Validation of Results obtained from MorFi

The second part of the research work was to find out if the Fiber Analyzer MorFi gives reliable results. Therefore 15 fiber samples have been analyzed with an alternative method that is described in chapter 4.3.4. Figures 33 – 47 show the results obtained from the alternative characterization method.

Figure 33 shows the analysis results for the fiber sample Modal with fiber titer 1.3 dtex and a starting length of 39 mm. The fiber did not contain any additives. An average fiber length of 789 µm was calculated.

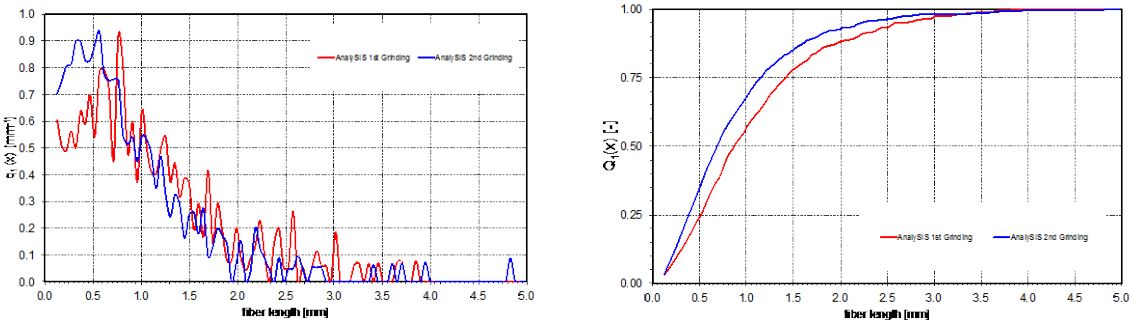

Figure 33: Modal 1.3 39 Bright comparative analysis

Figure 34 shows the results for the fiber sample Modal with fiber titer 1.3 dtex and a starting length of 39 mm. The sample contained TiO_2 for producing a satin finish. Based on the measured sum functions an average fiber length of 813 µm was calculated.

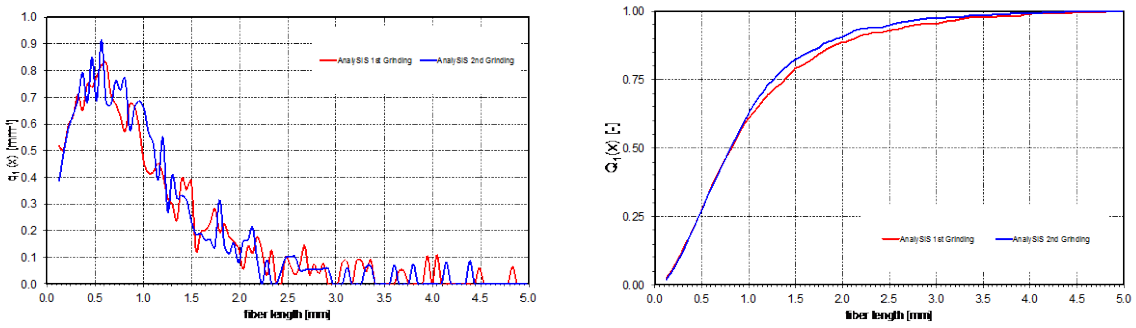

Figure 34: Modal 1.3 39 Dull comparative analysis

In Figure 35 the results for the colored modal fiber sample containing the pigment 6252 are shown. The fibers had a fiber titer of 1.5 dtex and a starting length of 39 mm. The analysis gave an average fiber length of 724 µm.

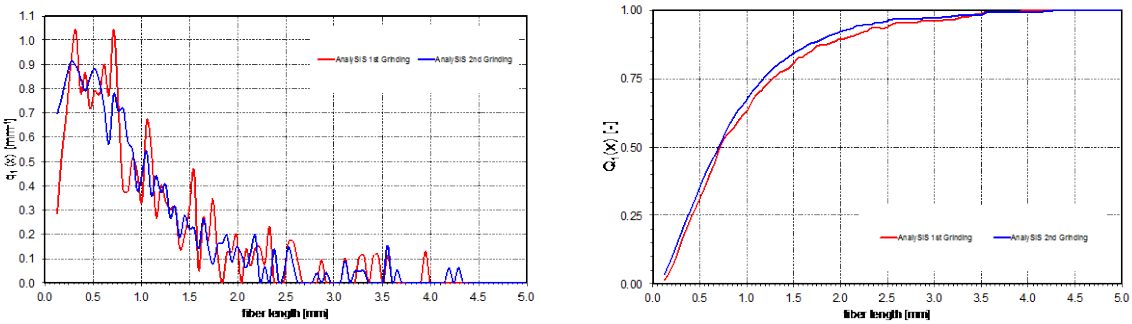

Figure 35: Modal 1.5 39 Bright 6252 comparative analysis

The results for the second colored Modal fiber sample with fiber titer 1.5 dtex and starting length 39 mm containing the pigment 6476, are summarized in Figure 36. An average fiber length of 808 µm was measured.

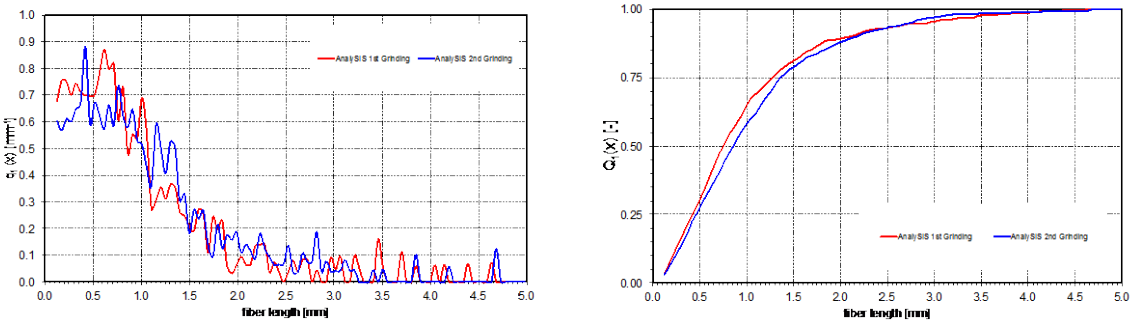

Figure 36: Modal 1.5 39 Bright 6476 comparative analysis

The third colored Modal fiber sample again showed the same fiber parameters like the other two samples and contained the pigment 6730. An average fiber length of 781 µm was calculated, again based on the sum functions. The results are summarized in Figure 37.

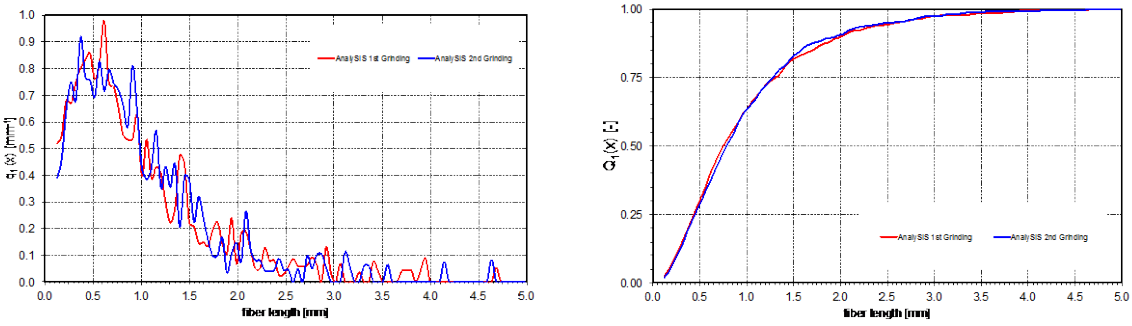

Figure 37: Modal 1.5 39 Bright 6730 comparative analysis

The heat protection fiber Modal FR was also analyzed with the alternative analysis method. Figure 38 shows the results. An average fiber length of 799 µm was measured.

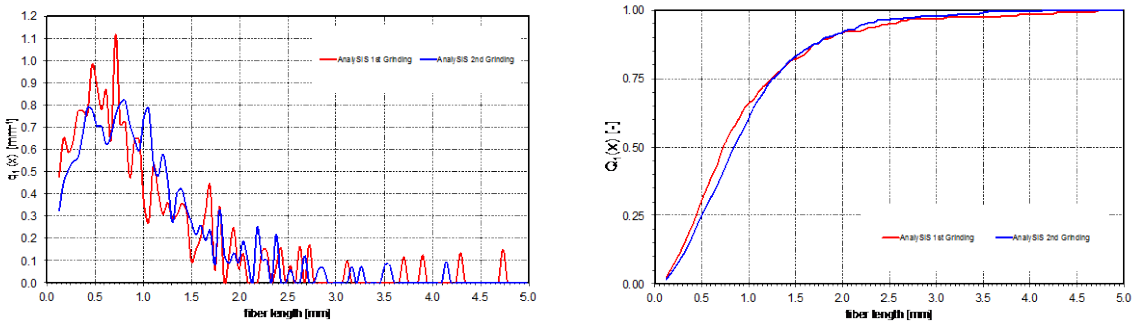

Figure 38: Modal FR 1.5 40 Dull comparative analysis

Seven Tencel fiber samples were also characterized using the comparative analysis method.

Figure 39 shows the results for measuring the fiber length after grinding for the fiber sample Tencel with 0.9 dtex fiber titer and 34 mm starting length. An average fiber length of 702 µm was calculated.

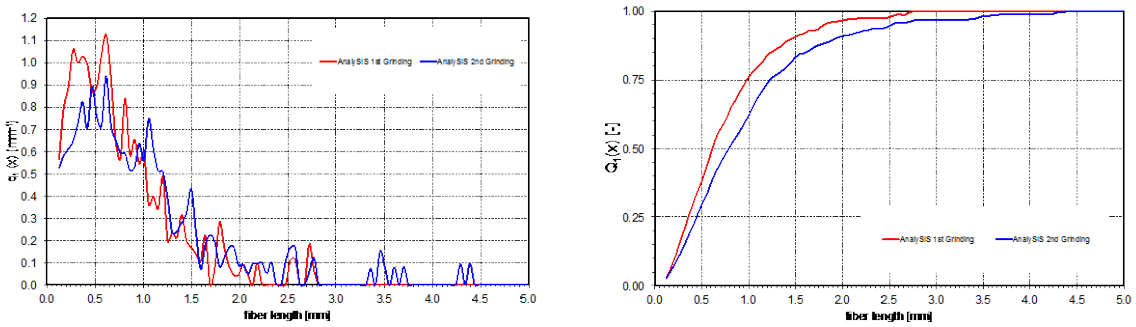

Figure 39: Tencel 0.9 34 Bright comparative analysis

Figure 40 gives a summary of the analysis of Tencel fiber samples with a fiber titer of 1.3 dtex and a starting length of 38 mm. The average fiber length of this Nonwoven fiber sample was calculated to be 857 µm.

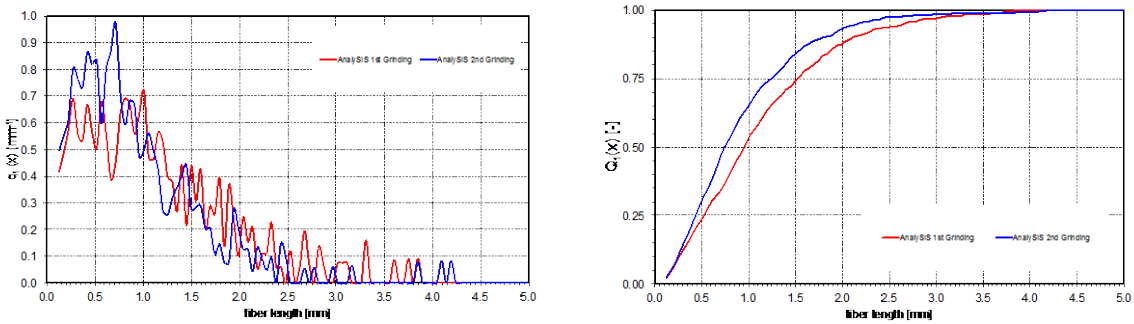

Figure 40: Tencel 1.3 38 Nonwoven comparative analysis

The Tencel Silicon fiber sample was also used for validating the results of MorFi System. An average fiber length of 721 µm was calculated. Figure 41 shows the results.

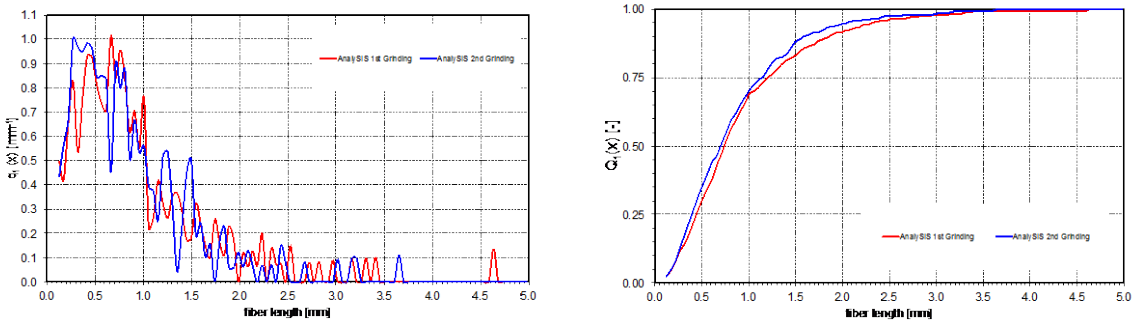

Figure 41: Tencel 1.7 6 Silicon comparative analysis

The comparative results for the fiber sample Tencel with a fiber titer of 1.7 dtex and a starting length of 38 mm are summarized in Figure 42. The analysis of this Nonwoven fiber sample gave an average fiber length of 707 µm.

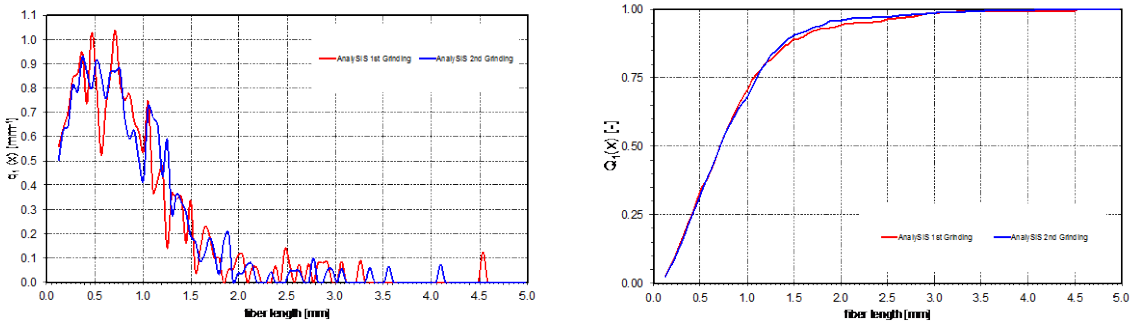

Figure 42: Tencel 1.7 38 Nonwoven comparative analysis

Figure 43 summarizes the results obtained from analyzing the Tencel fiber sample with fiber titer 1.27 dtex and starting length of 38 mm. As an average fiber length 945 µm were calculated.

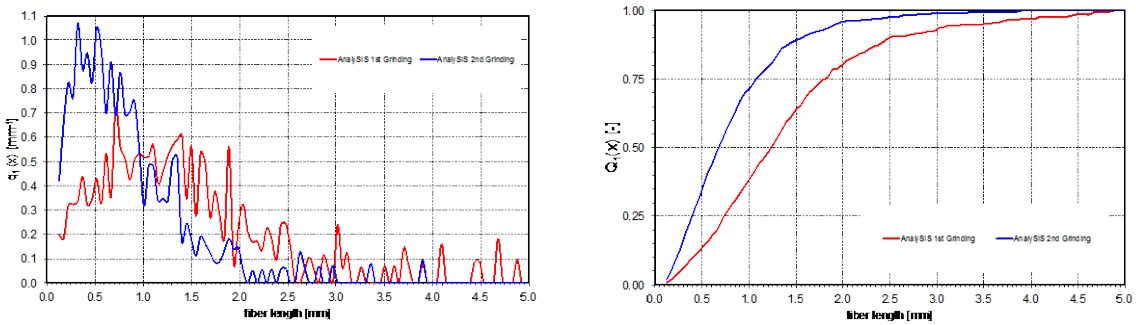

Figure 43: Tencel 1.27 38 Bright comparative analysis

The results for comparative analysis of the fiber sample Tencel with ZnO and a fiber titer of 2.5 dtex and 38 mm starting length are summarized in Figure 44. An average fiber length of 891 µm was calculated.

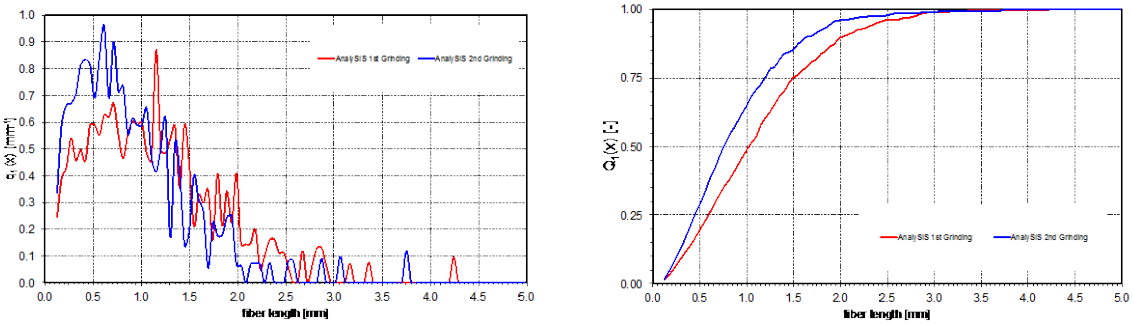

Figure 44: Tencel 2.5 38 ZnO

The second ZnO containing fiber sample was also analyzed with the alternative method. Figure 45 shows the results and based on the sum function an average fiber length of 1177 µm was calculated.

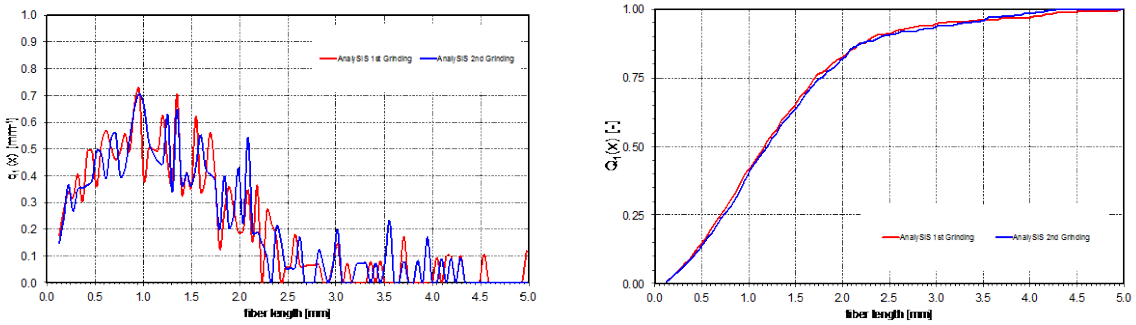

Figure 45: Tencel 6.7 60 ZnO comparative analysis

Two Viscose samples were also analyzed with the alternative method.

The Viscose fiber sample with a fiber titer of 1.3 dtex and a starting length of 39 mm gave an average fiber length of 786 µm. The results are summarized in Figure 46.

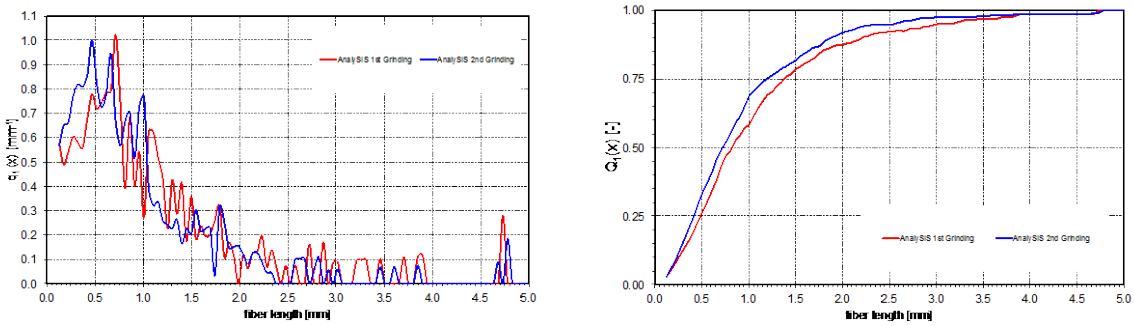

Figure 46: Viscose 1.3 39 Bright comparative analysis

The last sample analyzed for the comparative study was a Viscose fiber sample with 1.3 dtex fiber titer and 39 mm starting length. The fiber contained TiO_2 for deadening. For this sample an average fiber length of 790 µm was determined. Figure 47 shows the results.

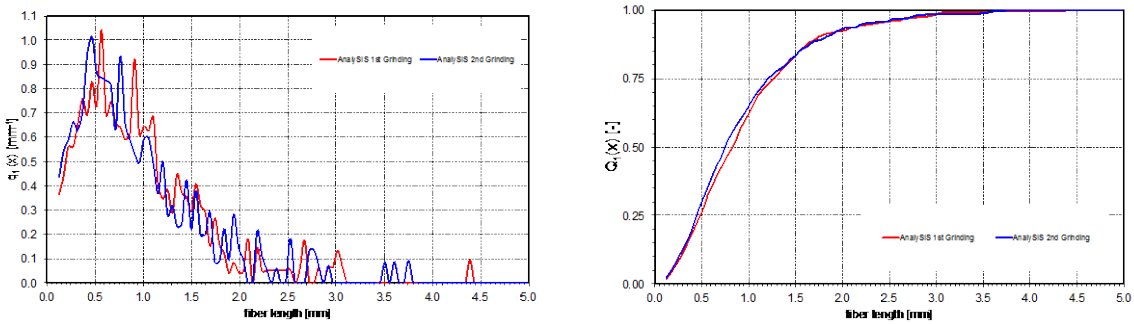

Figure 47: Viscose 1.3 39 Dull comparative analysis

Figure 48 summarizes the results for all comparative analysis.

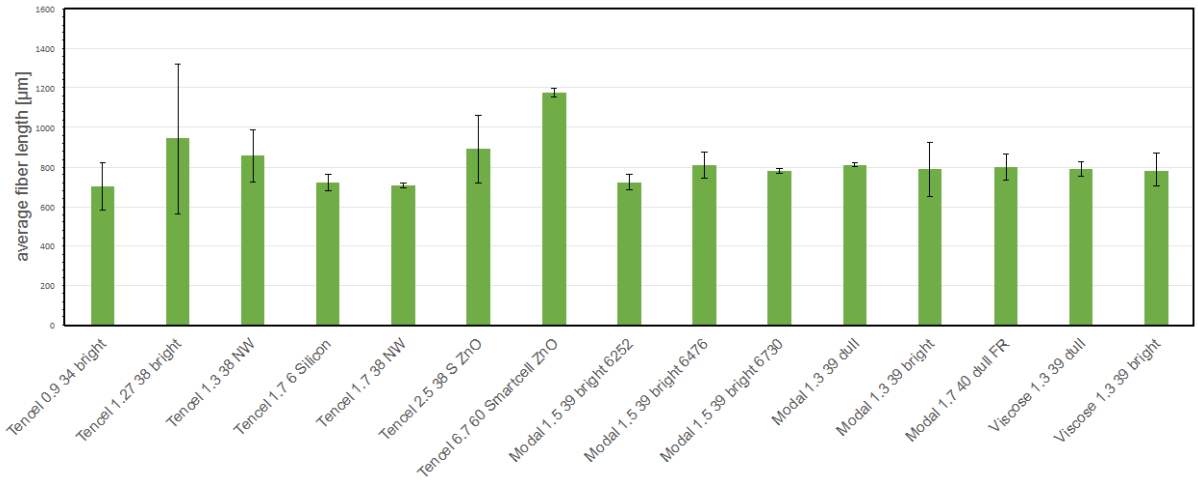

Figure 48: Comparative analysis summary

Table 5 shows the results for the comparative study and matches them with the results from MorFi analysis. Based on these results the difference between the two measurement methods was calculated.

Table 5: Summary of the comparative study

Fiber sample	Fiber length [µm]		Difference	
	MorFi	AnalySIS	[µm]	[%] of fiber length MorFi
Modal 1.3 39 Bright	789	809	20	2.5
Modal 1.3 39 Dull	813	861	48	5.9
Modal 1.5 39 6252 Bright	724	724	0	0
Modal 1.5 39 6476 Bright	808	839	31	3.8
Modal 1.5 39 6730 Bright	781	737	44	5.6
Modal FR 1.7 40 Dull	799	792	7	0.9
Tencel 0.9 34 Bright	702	729	27	3.8
Tencel 1.3 38 Nonwoven	857	795	62	7.2
Tencel 1.7 6 Silicon	721	697	24	3.3
Tencel 1.7 38 Nonwoven	707	767	60	8.5
Tencel 1.27 38 Bright	945	845	100	10.6
Tencel 2.5 38 Smartcell	891	901	10	1.1
Tencel 6.7 60 Smartcell	1177	1173	4	0.3
Viscose 1.3 39 Bright	786	804	18	2.2
Viscose 1.3 39 Dull	790	814	24	3.0
Average Difference	-	-	**32**	**3.9**

To characterize this average difference, a fiber sample (Modal 1.5 dtex 39 mm Bright 6901; ground using a 500 µm sieve) was prepared ten times for measuring on MorFi. These ten samples were then measured and the following results were received (Table 6).

Table 6: Statistic analysis of MorFi measuring process, determination of sample preparation error

Analysis Number	Average fiber length [µm]
1	535
2	523
3	518
4	531
5	521
6	538
7	522
8	555
9	538
10	517
Average fiber length of 10 samples [µm]	530
Standard deviation [µm]	12
Standard deviation [%]	2.3
Variance [μm^2]	142
2 x Standard deviation [µm]	24
3 x Standard deviation [µm]	36

The results for the ten measured samples are also plotted in Figure 49.

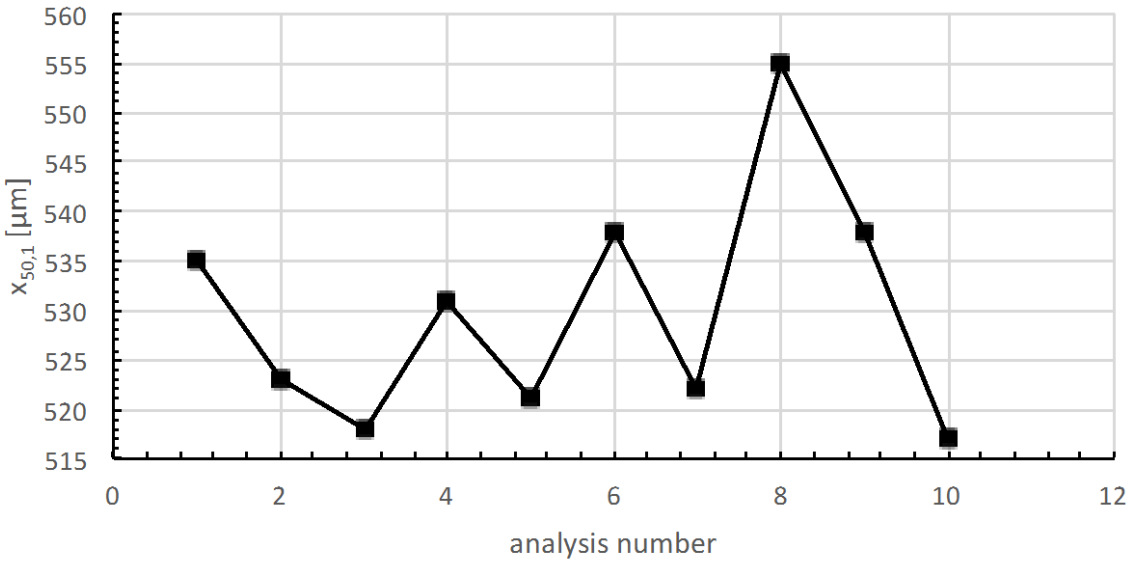

Figure 49: Statistic analysis MorFi measuring process

For the sake of completeness one single sample was measured 10 times with MorFi (fiber sample: Tencel FCP 10/300/M). The results for this series are summarized in Table 7 and plotted in Figure 50.

Table 7: Statistic analysis of MorFi measuring process, determination of repeatability

Analysis number	Average fiber length [µm]
1	293
2	295
3	296
4	293
5	292
6	297
7	296
8	297
9	293
10	292
Average fiber length [µm]	294
Standard deviation [µm]	2
Standard deviation [%]	0.7
Variance [µm^2]	4

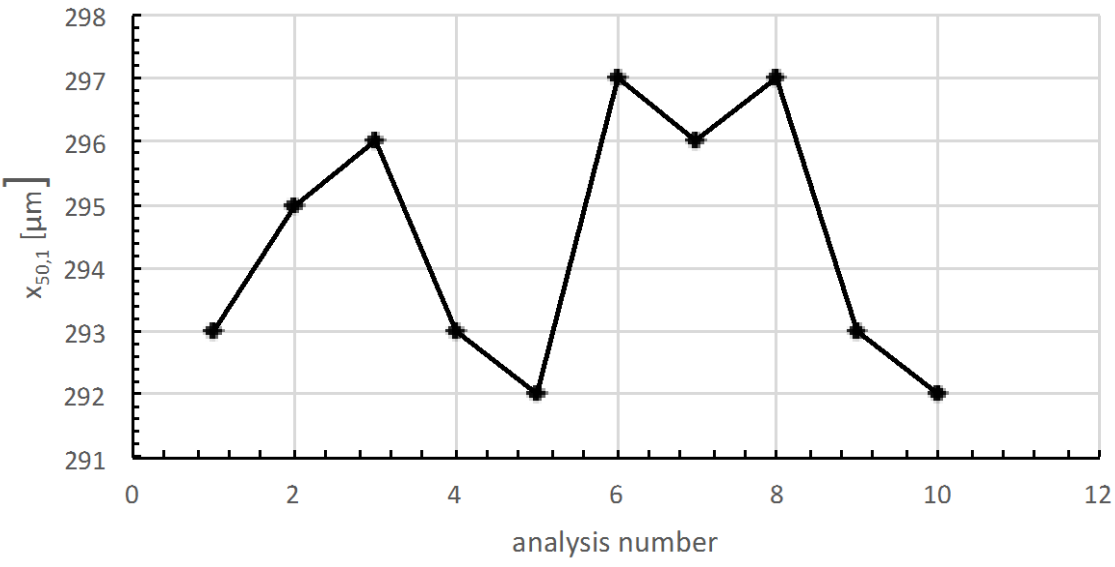

Figure 50: Statistic analysis MorFi measuring process

Based on the results from Tables 5, 6 and 7 it is possible to assume that the difference between the two analysis methods is not attributed to errors in measuring.

It seems that the difference is attributed to errors in sample preparation. Hence it is possible to say that MorFi's results are comparable to AnalySIS.

4.3 Development of a new and cheap Fiber Characterization Method

An essential part of this research work was the development of a new and cheap way to characterize the length of short fibers. The base for the development was optical light microscopy which is a commonly used method for characterizing fibers [26]. In the next chapters the workflow of evolving the new method is described.

4.3.1 Dry Dispersion

First experiments in the field of dispersing the fibers for a subsequent analysis were carried out with an Occhio Vacuum Disperser. Figure 51 shows the results for a two-step dispersion process, which means that an already dispersed sample was dispersed a second time. In a first step the sample was dispersed on a carrier. The sample on the carrier was then dispersed a second time onto a petri dish.

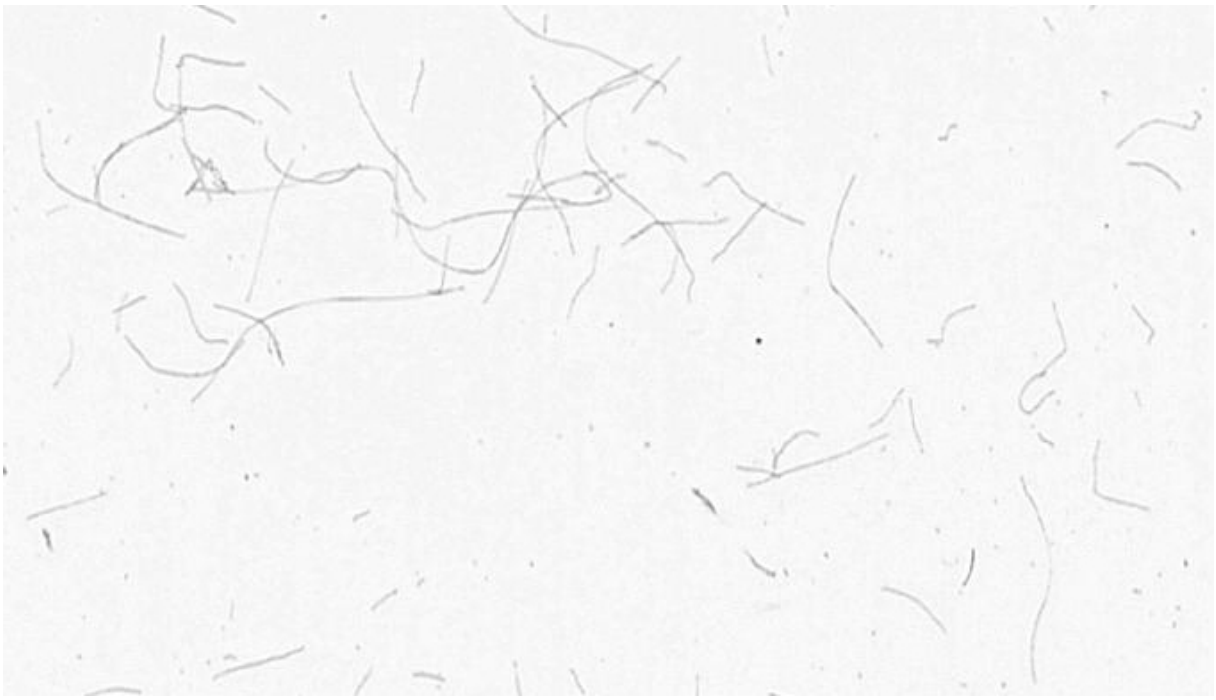

Figure 51: 2 step dry dispersion

These experiments showed that dry dispersion is not a useful tool for separating and dispersing short fibers, especially ground fibers which tend to have a very rough

surface and therefore tend to get stuck and build up fiber networks as imaged in Figure 51. Hence this dispersion method is not usable for an automated or semi automated characterization process. As a consequence experimental series using water as a dispersion media followed.

4.3.2 Wet Dispersion

Changing the dispersion media from air to water lowered the number of fiber networks and crossed fibers but brought new problems about. Using the Olympus Microscope System with it's moving Merzhäuser Table was not possible for recording sufficient large images due to problems with connecting the single images, which were recorded in the Multiple Image Alignment mode. It was only possible to record single images because the movement of the table obviously caused a movement of the fibers. Figure 52 explains the circumstance.

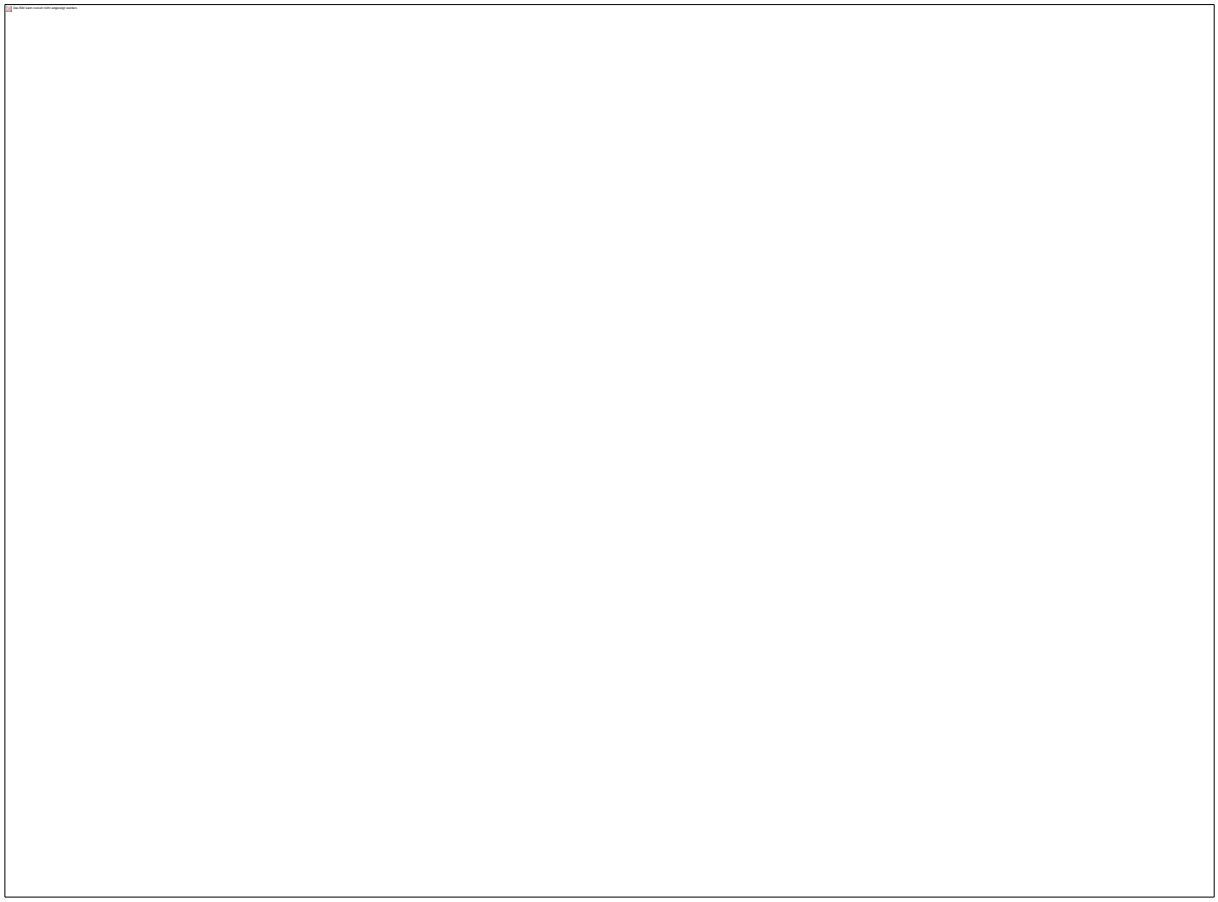

Figure 52: Wet dispersed fiber sample recorded with an Olympus microscopy system

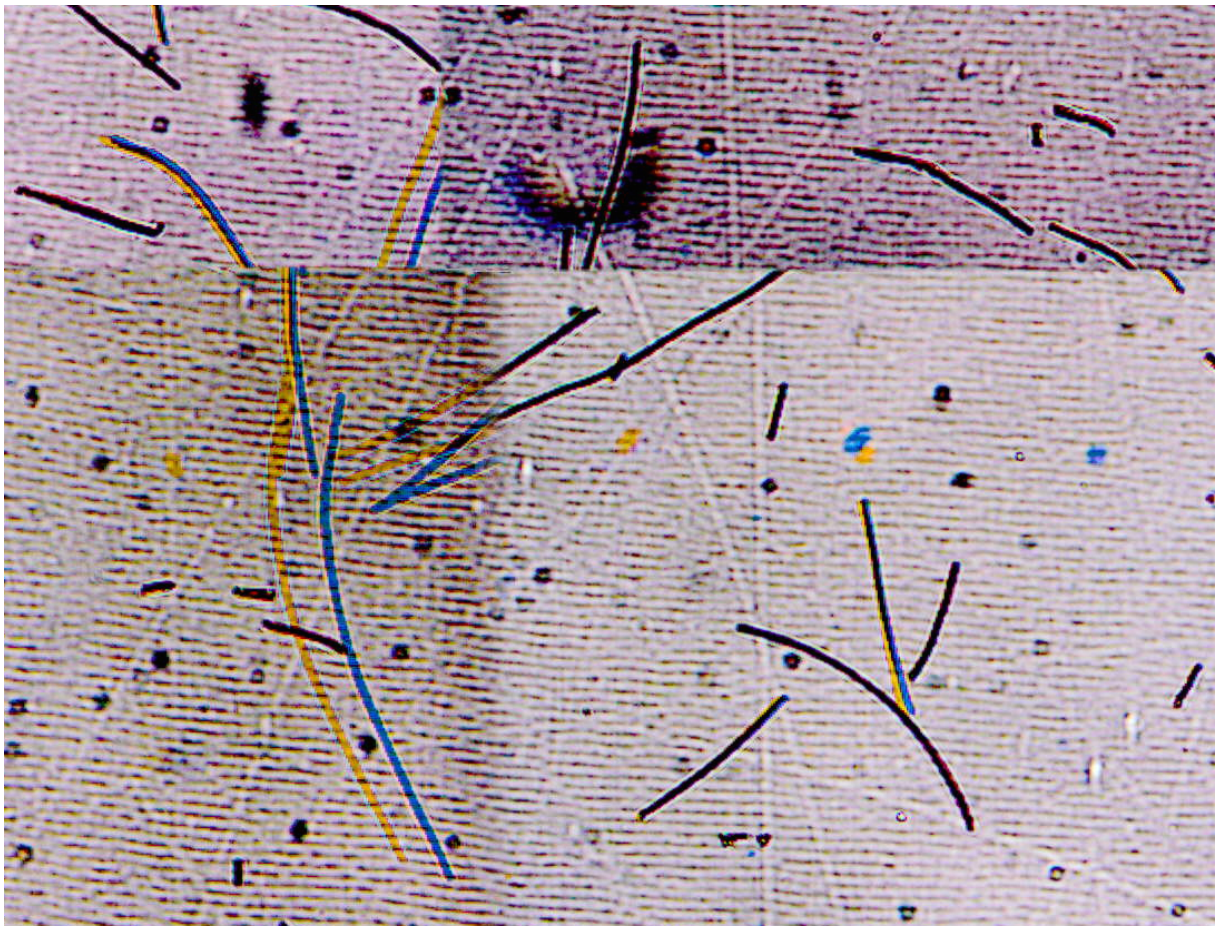

Figure 53: Enlargement of an image section of Figure 52

As shown in the Figure 52, the microscopy system is not able to create a whole, analyzable image. Figure 53 shows in detail the problems. Due to the movement of the Merzhäuser Table the fibers are always moving too. This movement is the reason for not fitting images, cropped fibers which would be analyzed as two separate fibers and moving fibers which are not able to be detected as one single object. Due to this fact, a characterization method using a microscopy system does not seem to be a suitable solution for characterizing ground fibers. For further experimental series only the microscopy software AnalySIS from Olympus was used but not the microscopy system itself.

4.3.3 Image Recording System

As Bartl et al mentioned, a high resolution photo scanner can be used as a powerful tool for recording images of dispersed fibers [39]. Based on this work the microscopy system was replaced by an Epson V500 Photo scanner, which had a resolution of 4

µm/pixel. Introducing this image recording system showed both, advantages and disadvantages. From then on it was possible to record bigger images in a faster way. However a photo scanner has to use a very strong light source for creating images of films or slide positives and negatives. This strong light source caused problems when measuring white, non-colored fibers; images of colored fibers were possible.

4.3.4 Contrast Problems

Figure 54 shows a scanner image of white fibers. The contrast between fiber and background became too low due to the strong light source of the scanner. As it is possible to see in the middle of the image, the microscopy software cannot distinguish between fiber and background due to brighter regions in the fiber and separates this object into several autonomous objects. This would end up in a higher number of shorter particles than in reality and in an average fiber length that is lower than the real one. It was attempted to solve this problem by changing the gray tone border (value which tells the software what is a fiber and what is background) between background and fiber. Even a change of the threshold values did not improve the contrast. Figure 55 and Figure 56 show the results for these attempts.

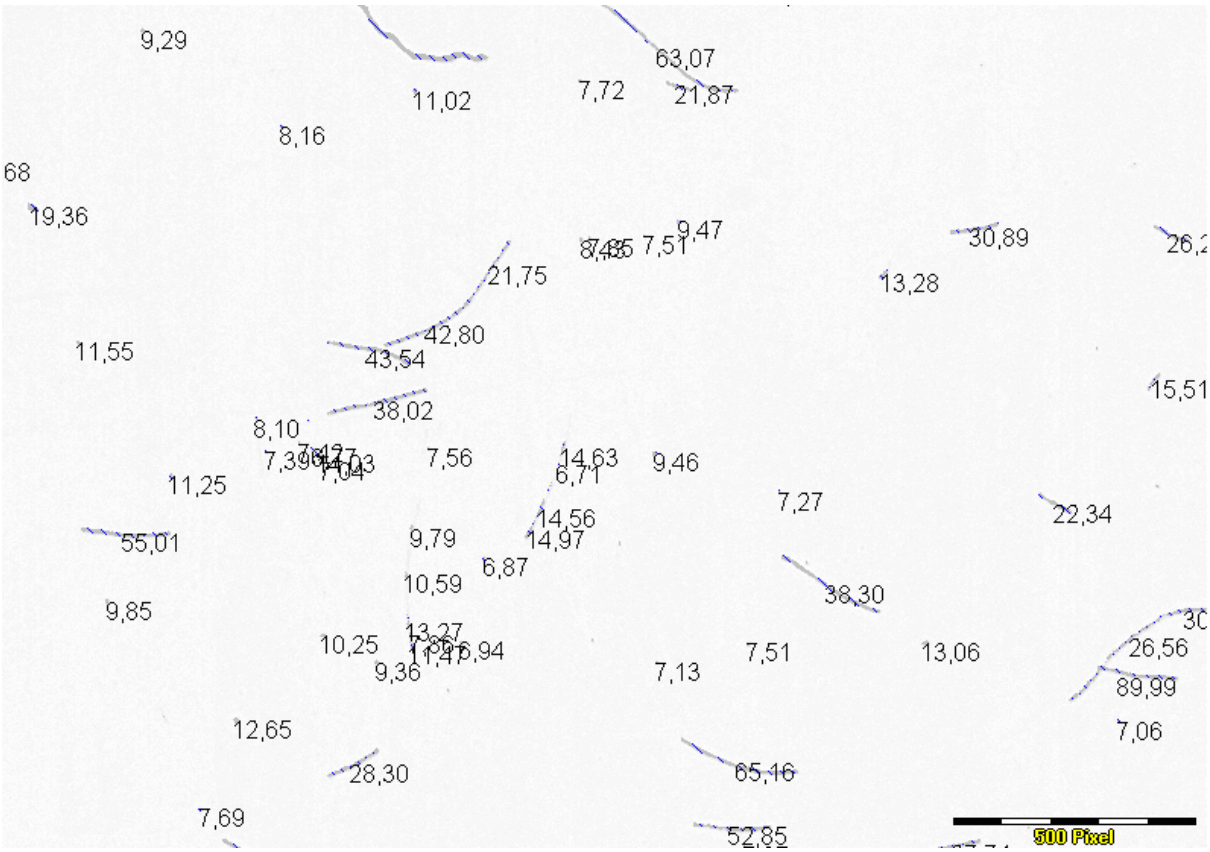

Figure 54: Attempt to analyze white fibers

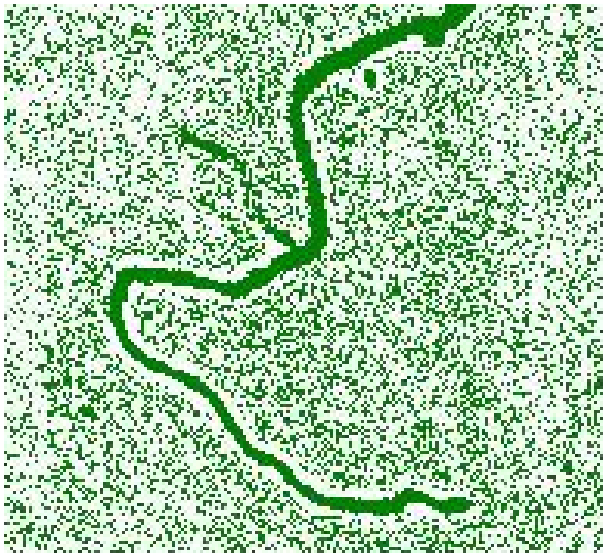

Figure 55: Increasing the gray tone border

Increasing the gray tone border leads to the effect figured in Figure 55. The fiber becomes bigger than it is and parts of the background are recognized as particles. Summarized, analysis is not possible in a correct way. Figure 56 shows what happens when analyzing an image using this grey tone border conditions.

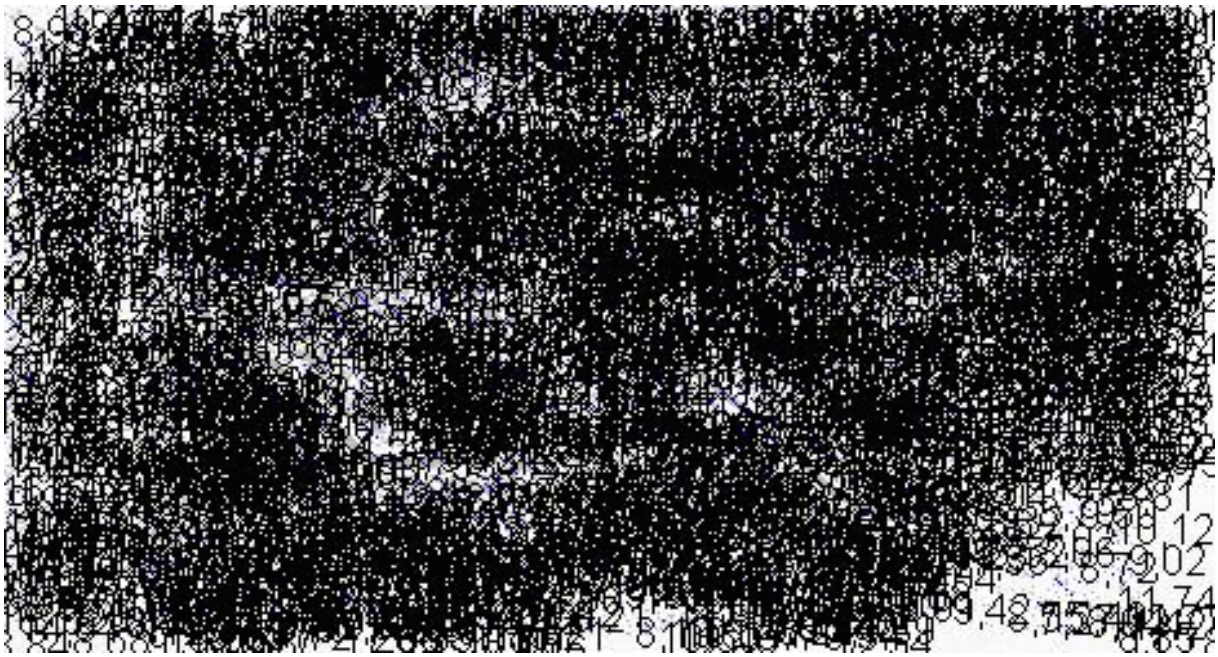

Figure 56: Increased grey tone border

As mentioned, the software identifies parts of the background as particles and correct analysis is not possible anymore.

In a first approach it was tried to improve the contrast between fiber and background using digital technologies implemented in the software of the photo scanner.

Figure 57 and Figure 58 show the results for the digitally arranged sample images.

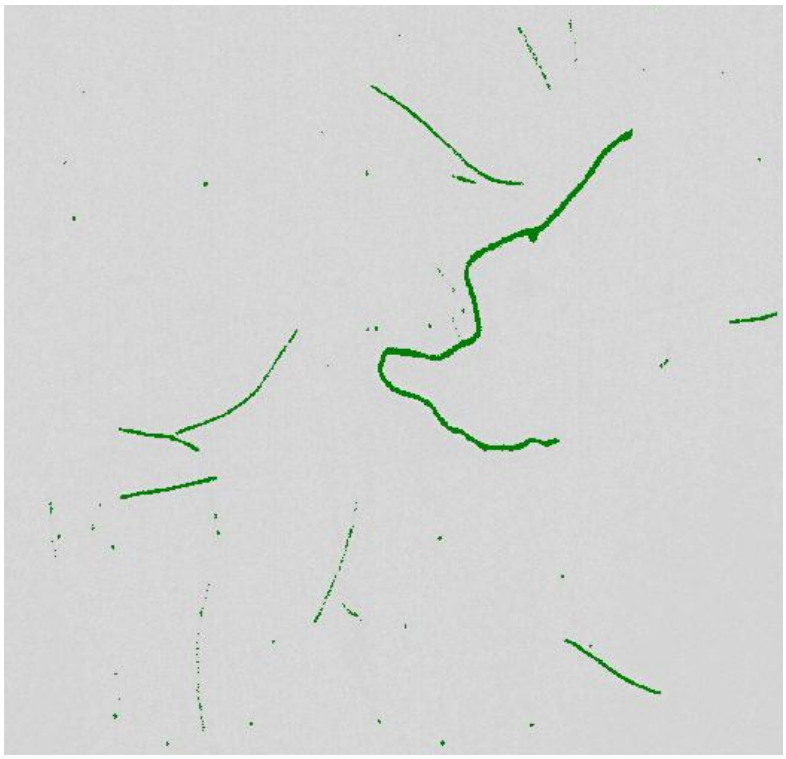

Figure 57: Digitally arranged sample image, normal grey tone border

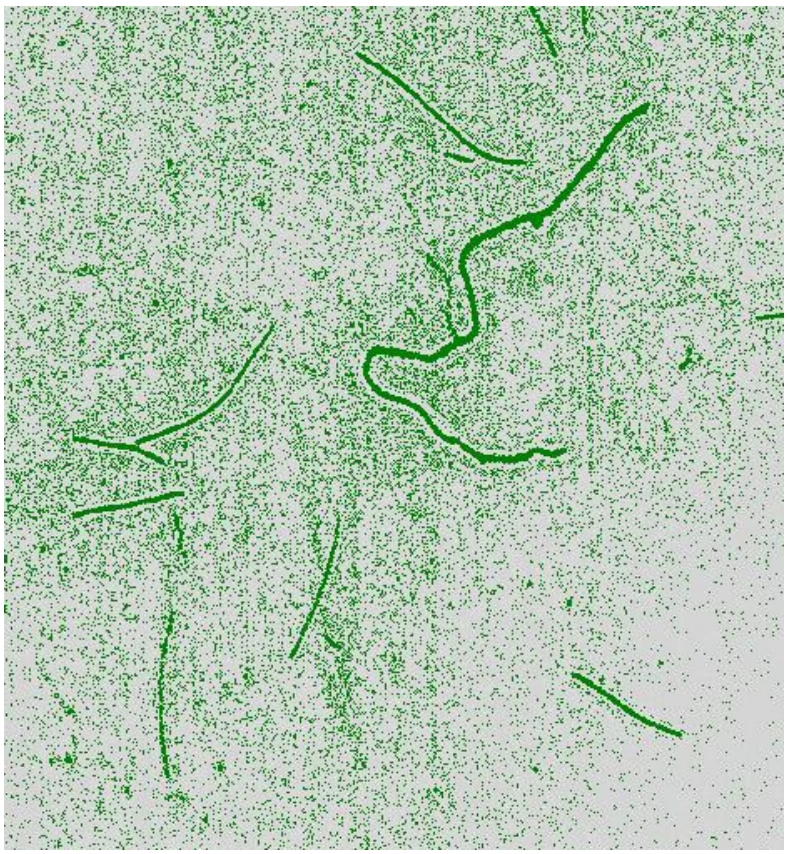

Figure 58: Digitally arranged sample image, increased grey tone border

Again the same problems occurred. Using the normal grey tone border ended up again in “cutting” fibers. Increasing the grey tone border caused the effect presented in Figure 58.

Due to these facts another way for enhancing the contrast between fiber and background was introduced. A reactive dye was used to color the fibers dark blue/ black (further information in experimental part, chapter 3.5). The results for recording colored fiber samples are presented in Figure 59.

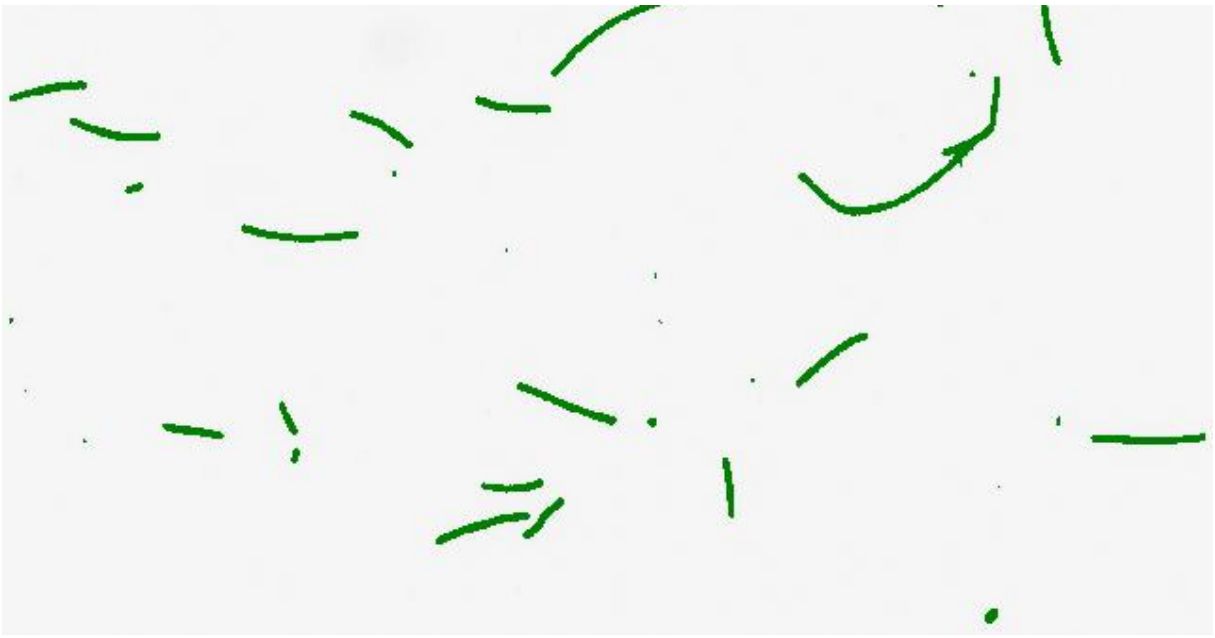

Figure 59: Colored fibers

Coloring the fibers made it possible to define a threshold value feasible to distinguish the background from the fibers. Thus it was possible to carry out a comparative analysis with MorFi (chapter 4.2). Crossed fibers still had to be excluded manually and image analysis was performed using the AnalySIS software.

4.3.5 Software

The most expensive part of the characterization equipment was the microscopy software AnalySIS from Olympus. As an alternative the software Scientific Counter was introduced. To check weather Scientific Counter gives reliable results, a fiber sample (black Modal fiber, ground using a 500 µm sieve of circular mesh) was

analyzed with MorFi, Scanner and AnalySIS and Scanner and Scientific Counter. The results are summarized in Figure 60.

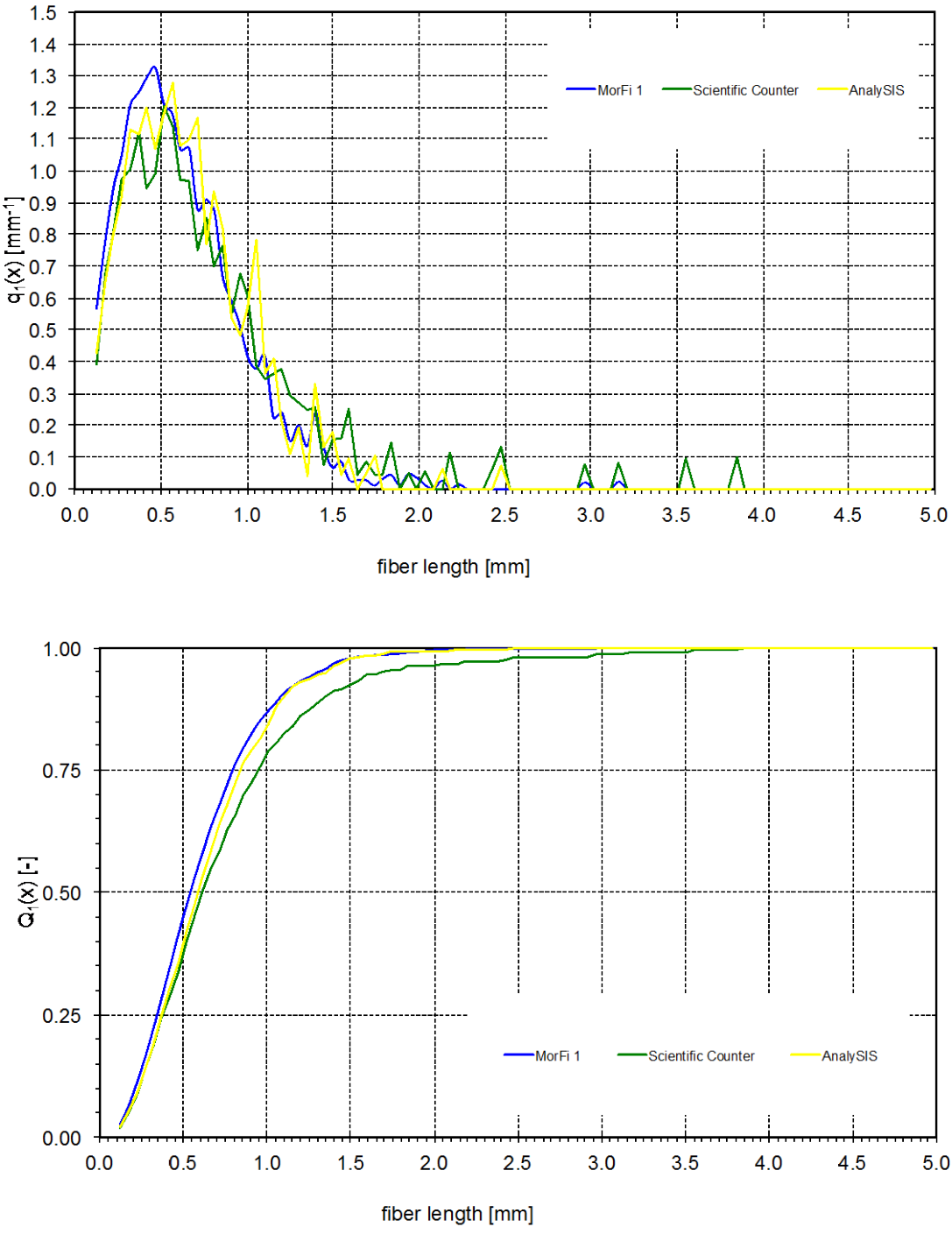

Figure 60: Comparative study between MorFi, AnalySIS and Scientific Counter

Based on this data it is obvious that Scientific Counter gives reliable results. The small differences may be caused by errors when preparing the samples and by

differences in the analysis algorithms of the image analysis software systems (chapter 4.2 part statistic analysis MorFi).

4.3.6 Exclusion of Crossed Fibers

A very important part of the development of the new fiber characterization method was to exclude or reduce crossed fibers because they cannot be analyzed properly. A few mathematical approaches based on commonly used particle parameters were investigated but until now no satisfying results were obtained. Due to this fact experiments with different fiber concentrations and during the further procedure with different dispersion fluids were carried out. For these experimental series the Bright fiber sample Tencel with 1.27 dtex fiber titer and 38 mm starting length was used.

Figures 61 - 68 show the results for the concentration experiments.

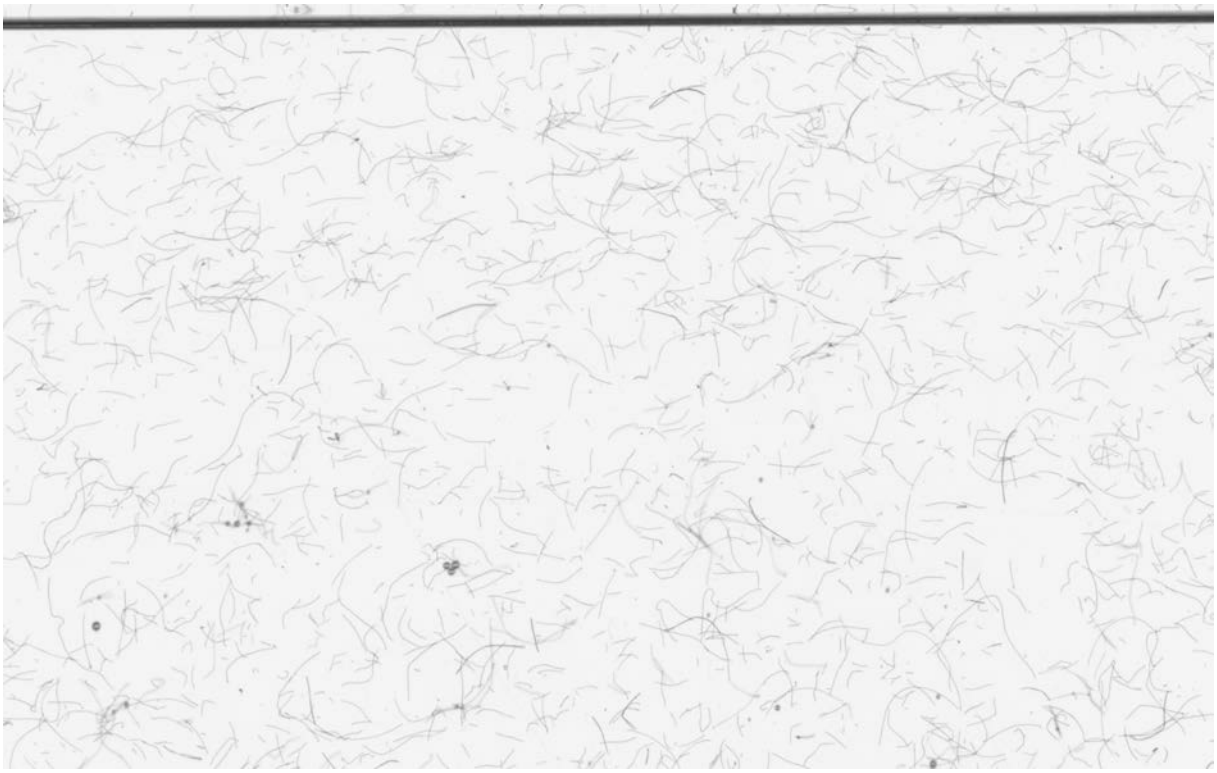

Figure 61: 90 mg/l

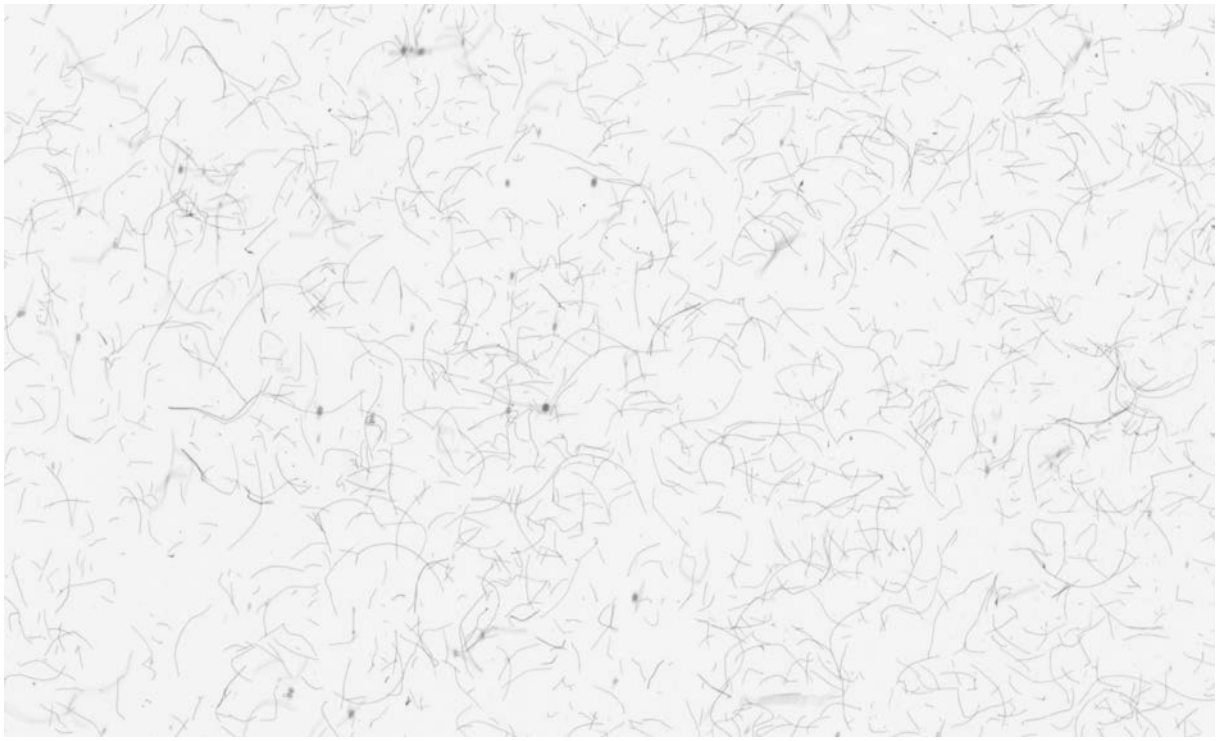

Figure 62: 70 mg/l

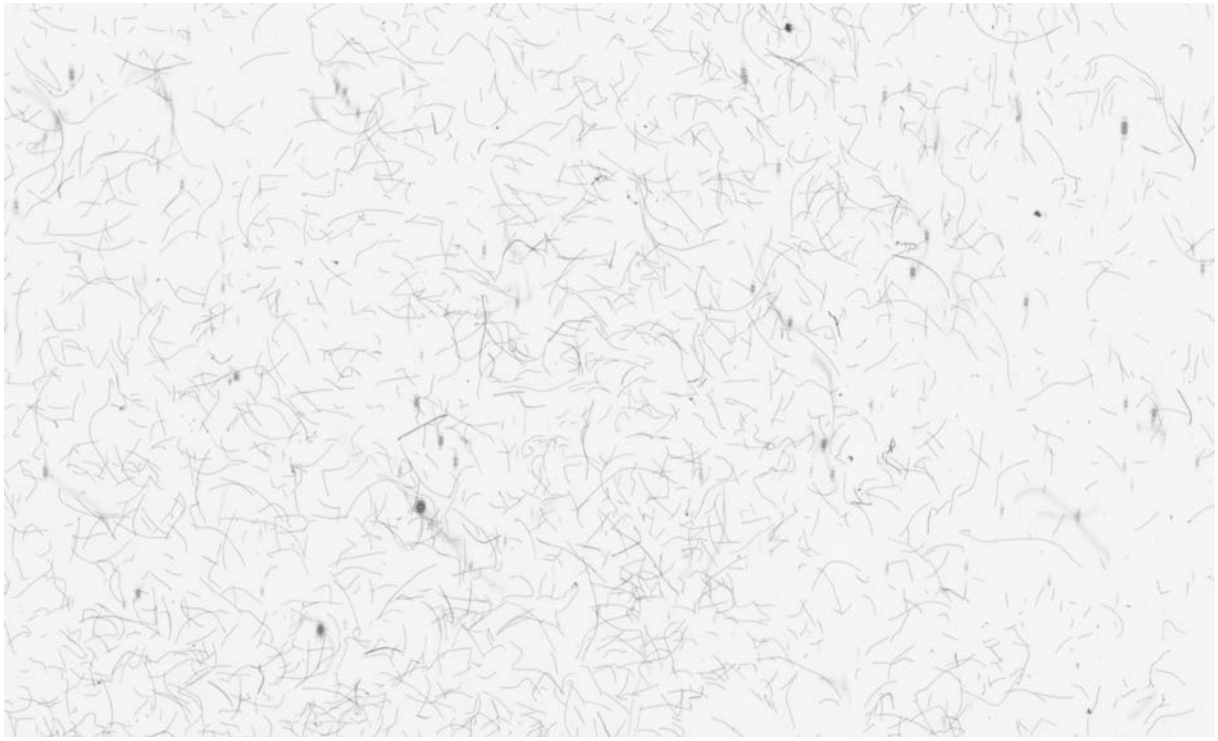

Figure 63: 54 mg/l

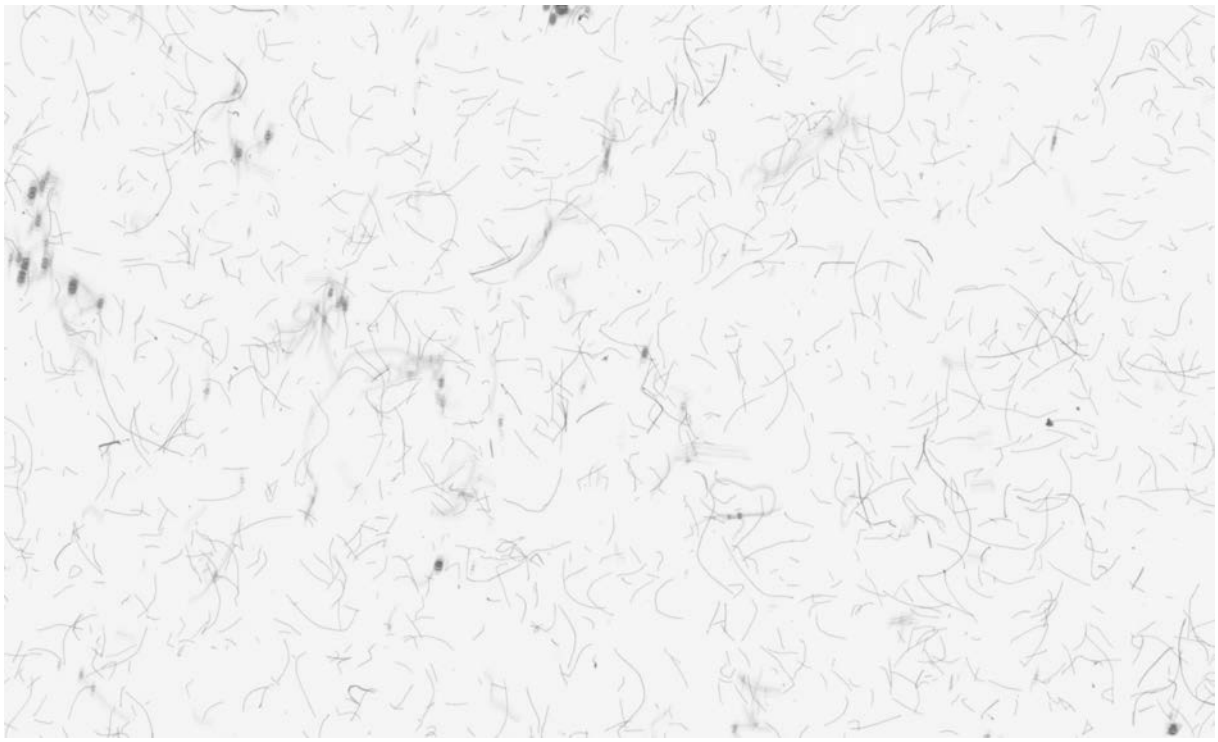

Figure 64: 42 mg/l

Figure 65: 18 mg/l

Figure 66: 10 mg/l

Figure 67: 5 mg/l

Figure 68: 2.5 mg/l

Table 8 summarizes the results.

Table 8: Summarized results concentration screening

Concentration [mg/l]	Suitable y/n
90	No
70	No
54	No
42	No
18	No
10	No
5	No
2.5	Suitable (13 crossed objects)

The experiments showed that the most useful fiber concentration is 2.5 milligrams per liter (50 ml of dispersion were prepared). This concentration still caused crossed fibers; hence experiments with different dispersion fluids were carried out. The results are figured in Figure 69 – 74.

Figure 69: Distilled water

Figure 70: Electrolyte solution (80 g/l NaCl, 40 g/l Na_2CO_3 and 2 g/l NaOH)

Figure 71: Water with Neutracon (surface active spezies)

Figure 72: PEG : Water 50:50

Figure 73: PEG 400

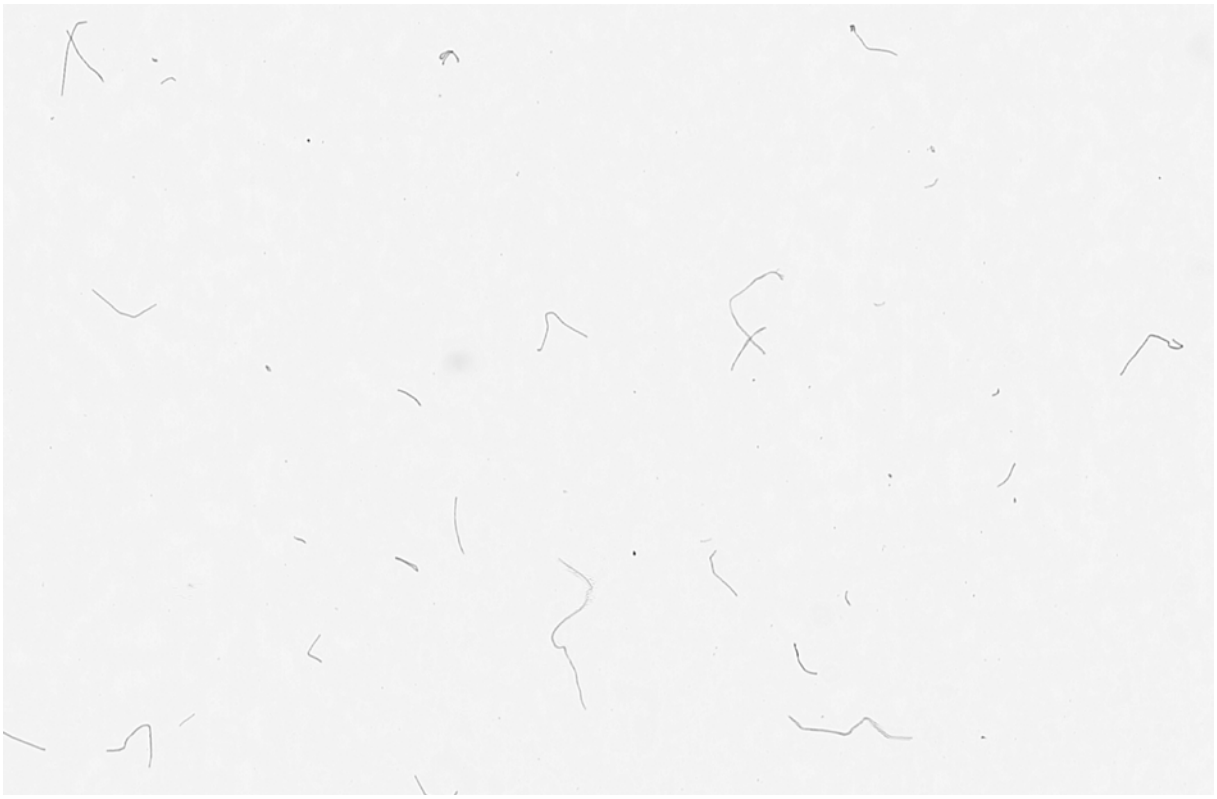

Figure 74: Water with PVA 80% hydrolyzed (surface active substance)

Table 9 summarizes the results.

Table 9: Summarized results dispersion media screening, 2.5 mg/l concentration

Dispersion fluid	Suitability (area: ~68 x 120 mm)
Distilled water	No (more than 32 crossed objects)
Electrolyte solution	No (16 crossed objects)
Water with Neutracon	No (more than 60 crossed objects)
PEG 400:Water 50:50	No (13 crossed objects but viscosity becomes to low wherefore the positive properties of PEG 400 get lost)
PEG 400	Quite good, but still 13 crossed objects
Water with PVA 80% hydrolyzed	No (22 crossed objects)

Due to the results from the dispersion media screening and the concentration screening it was necessary to lower the concentration again. Two pre-experiments were carried out with water. Both samples contained about 0.6 mg/l fibers (prepared via dilution series). Figure 75 and Figure 76 show the results.

Figure 75: Pre-experiment with water and 0.57 mg/l sample

Figure 76: Pre-experiments with water and 0.56 mg/l sample

These pre-experiments showed that a fiber concentration of 0.5 mg/l could be a feasible way (in these samples only one respectively two crossed objects could be found). Using this concentration with PEG 400 as a dispersion fluid made it possible to disperse the fibers without any crossing. Figure 77 presents the results.

Figure 77: Experiment with PEG 400 and a fiber sample concentration of 0.5 mg/l

The proper concentration was regulated via a dilution series. This approach also ensures the correct sample taking.

The dilution series was carried out following Figure 78. With this last step, the development of the new characterization method was finished. For other fiber sample types a first approach for the amount of fibers for the dilution series can be calculated based on the bulk density, the fiber titer and the starting length. Via a coefficient, which is calculated from the parameters mentioned before of the fiber sample Tencel 1.27 dtex 38 mm Bright and the same parameters of the unknown sample a first weighted sample can be calculated. A suitable weighted sample has to be found via tests.

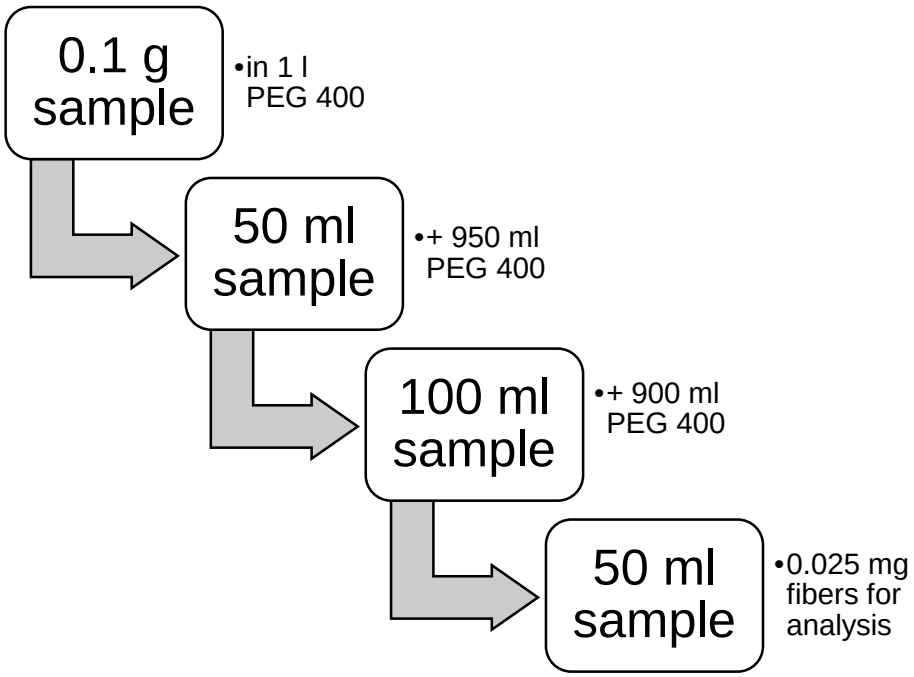

Figure 78: Dilution series

4.3.7 First Test

Finally the new characterization technique was checked for a representative fiber sample. For checking it the total procedure was carried out analyzing the colored fiber sample Tencel 1.27 dtex 38 mm Bright. Figure 79 summarizes the individual steps of the procedure.

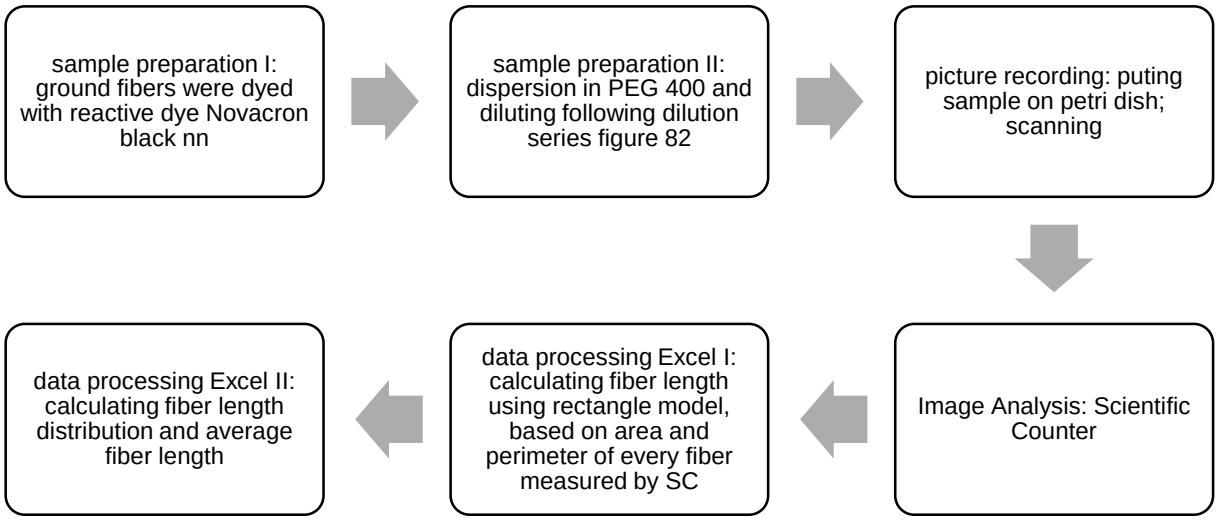

Figure 79: Individual steps of new developed fiber characterization technique

The results for testing the new method are plotted in Figure 80 and compared with a MorFi analysis of the used sample.

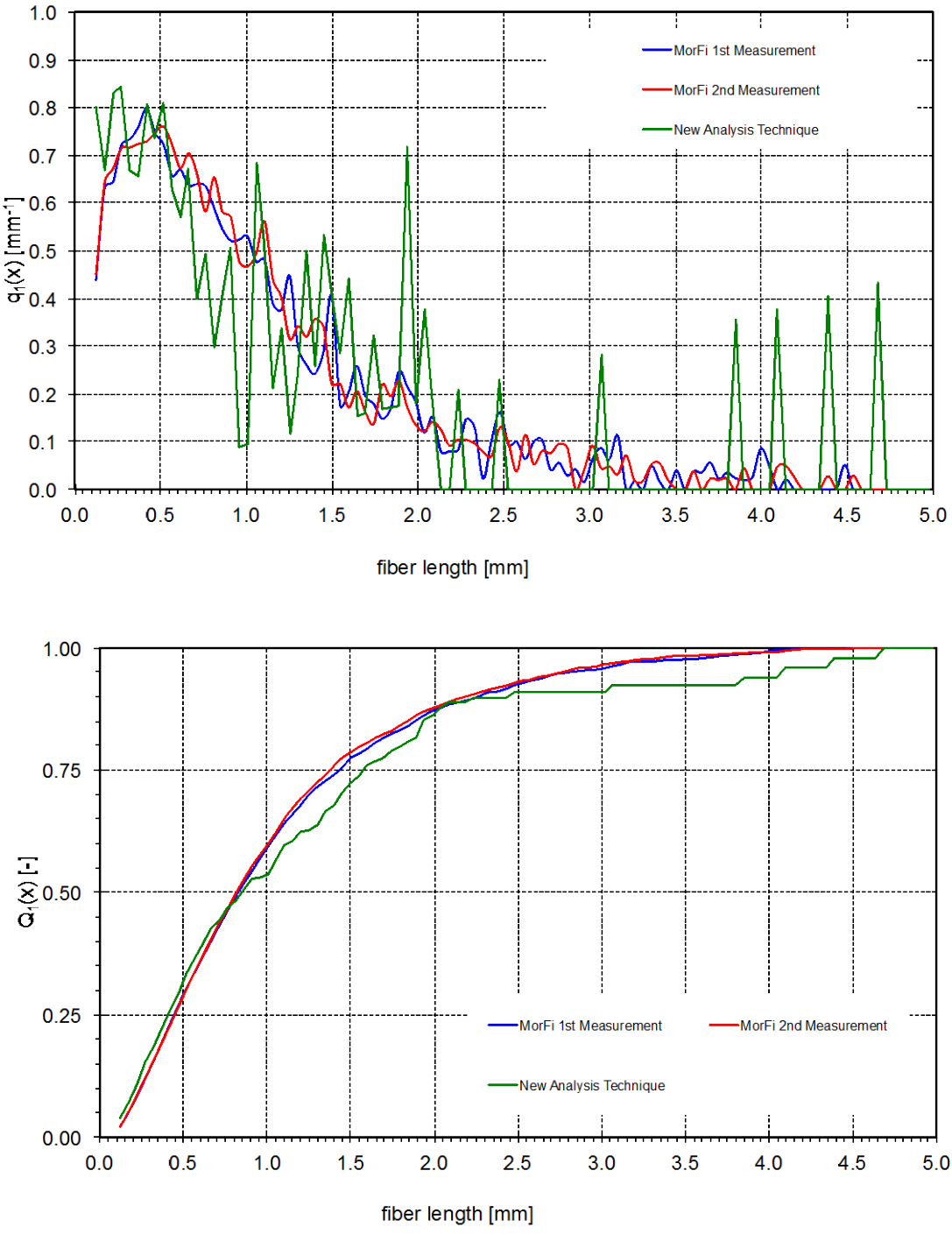

Figure 80: Comparison of new analysis technique with MorFi

Table 10 summarizes the results for testing the new method.

Table 10: Summary of results testing the new analysis technique (NAT)

Analysis Method	Fibers measured	Average fiber length [µm]
MorFi	~ 10.000	824
NAT	~ 300	785

A drawback of Scientific Counter is the limitation of the image size (2 MB). Larger images would be required to count a larger number of individual fibers. DatInf announced an update for Scientific Counter, which should be available soon.

To gain access to larger images (they are necessary to get enough fibers for analysis because the number of fibers in the diserpersion is very low due to the very high dilution rate which is needed to oppress crossed objects), bigger ones were divided using the image processing software Gimp. Gimp is a very simple but powerful tool, which is able to separate images into smaller ones without the loss of quality. After dividing the images into several smaller ones, Scientific Counter was able to analyze them. Due to this very time consuming limitation only around 300 fibers were analyzed. Nevertheless it seems that the new characterization technique produces reliable results. As already shown in chapter 4.2 the difference of 39 µm seems to be caused by errors in sample taking and different software algorithms.

4.4 Bulk Density as a Characteristic for Quality of Fiber Grinding

The bulk density is a commonly used characteristic for the quality of fiber collectives in fiber industry. Hence from all investigated samples the bulk density was determined to check if this parameter could be used as a quality tool for checking the grinding process. Figure 81 shows the results for these determinations and Table 11 summarizes them.

Table 11: Summary bulk densities grinding experiments (standard grinding conditions (Table 2), 2 mm circular mesh sieve)

Fiber sample	Bulk density [g/l]
Modal 1.3 39 Bright	20.86
Modal 1.3 39 Dull	19.09
Modal 1.5 39 Bright 6252	25.55
Modal 1.5 39 Bright 6476	20.00
Modal 1.5 39 Bright 6730	25.34
Modal FR 1.7 40 Dull	23.67
Tencel 0.9 34 Bright	17.95
Tencel 1.27 38 Bright	18.34
Tencel 1.3 38 Nonwoven	19.83
Tencel 1.7 38 Nonwoven	25.39
Tencel 1.7 6 Silicon	29.96
Tencel 2.5 38 Smartcell	40.25
Tencel 6.7 60 Smartcell	78.71
Viscose 1.3 39 Bright	23.98
Viscose 1.3 39 Dull	19.80
Viscose 17 100 Dull	90.92
Viscose 3.3 60 Bright	27.62
Viscose 4.2 60 Dull Nonwoven	47.44
Viscose 5 120 Bright	56.75

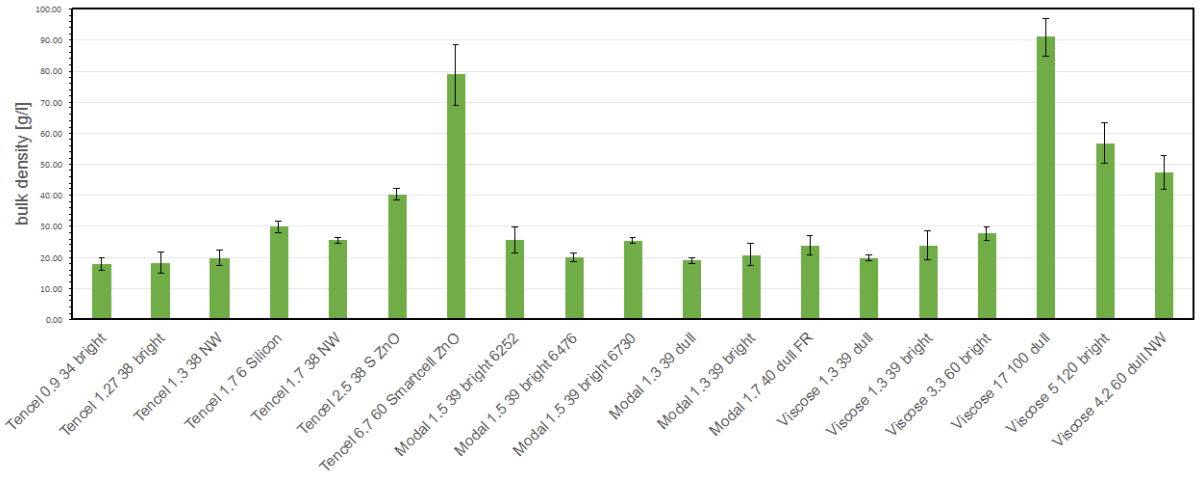

Figure 81: Bulk density of the ground fiber samples (standard grinding conditions (Table 2), 2 mm circular mesh sieve)

5 Discussion

5.1 Grinding Process

An important part of this work was to find out more about the influence of different fiber and process specific parameters on the resulting fiber length in a fiber grinding process. Generally each fiber sample showed a different behavior concerning the grinding process. In the next chapters the influence of these parameters are discussed.

5.1.1 Influence of Fiber Diameter and Fiber Starting Length

It has to be mentioned that for this study no set of adequate fiber samples has been available. In particular fiber samples of different starting lengths also showed different diameters and vice versa. Thus it is difficult to correlate the results to a specific parameter. Figure 82 and Figure 83 plot the relationship between starting length/diameter and average fiber length after grinding.

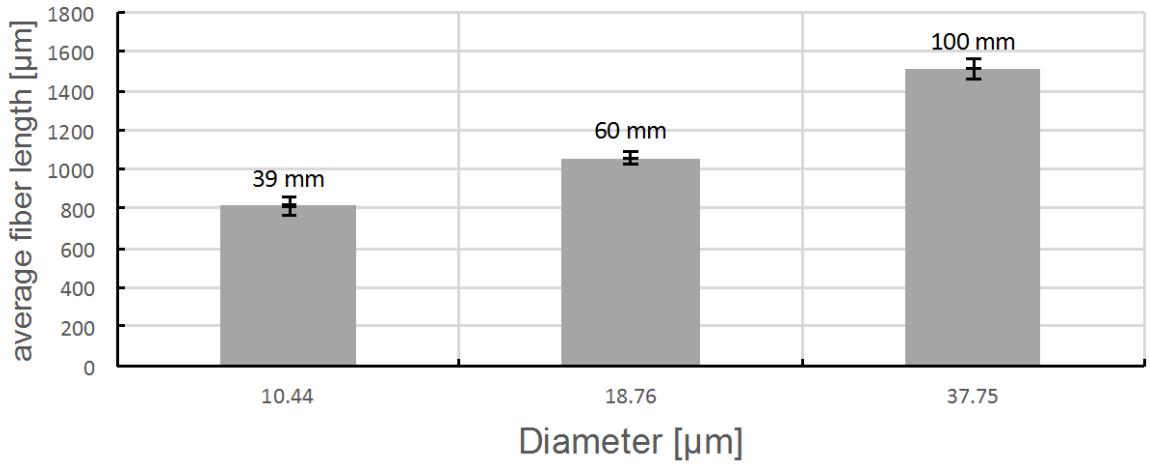

Figure 82: Relationship between fiber diameter/starting length and average fiber length after grinding for Dull fiber samples

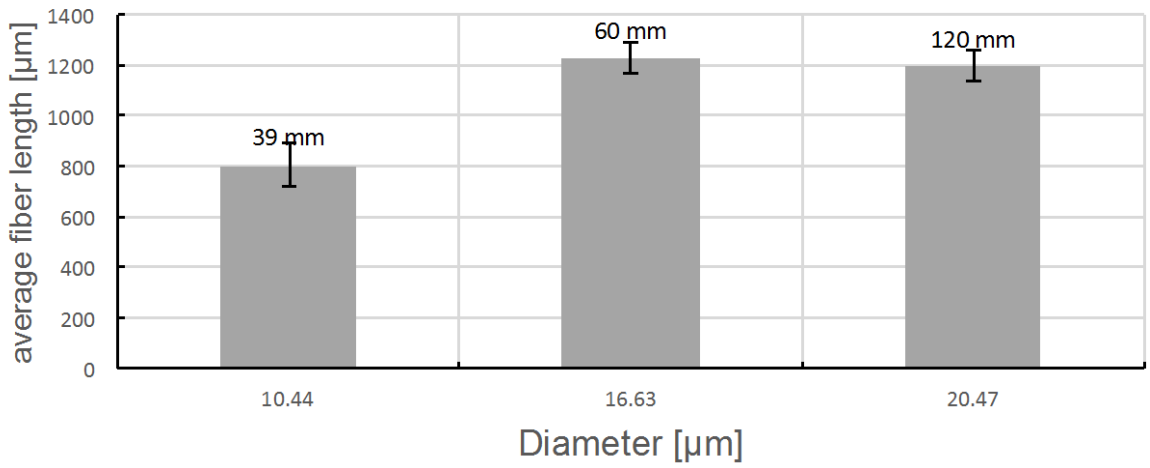

Figure 83: Relationship between fiber diameter/starting length and average fiber length after grinding for Bright fiber samples

It can be concluded that increasing the fiber length and diameter leads to a larger average fiber length. Four Tencel samples with very similar or even with equal starting lengths were also investigated to find a relationship between fiber diameter and the resulting average fiber length after the grinding process.

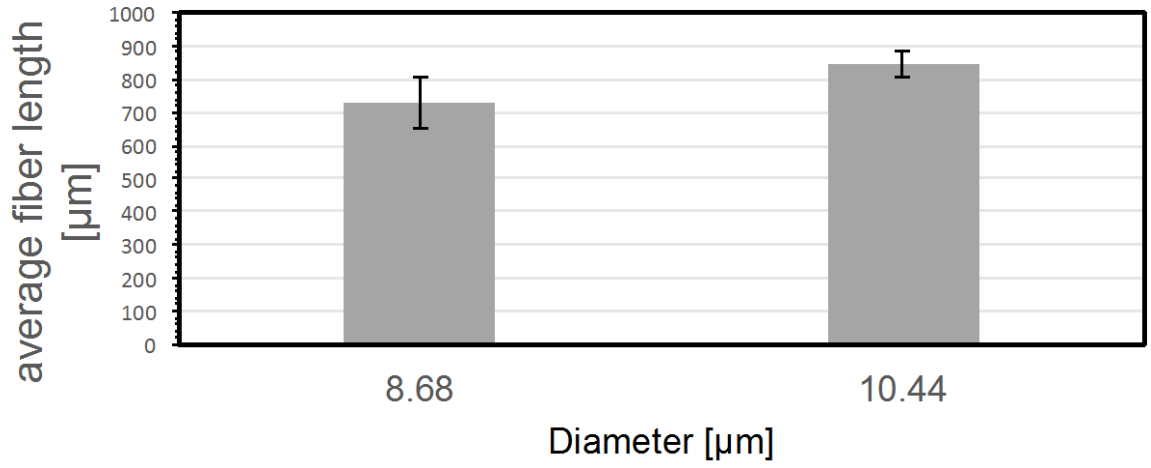

Figure 84: Influence of fiber diameter, samples Tencel Bright

As presented in Figure 84 the experiments with Bright Tencel fibers showed that a larger fiber diameter (equal to a larger fiber titer) causes a larger average fiber length. It has to be considered that the sample with smaller diameter showed a slightly shorter starting length.

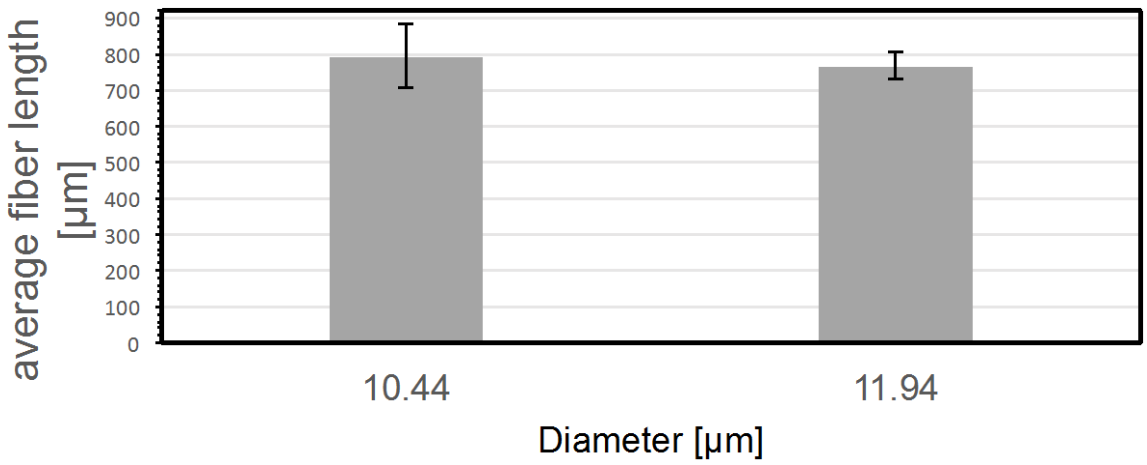

Figure 85: Influence of fiber diameter, test samples Tencel Nonwoven (10.44 µm and 11.94 µm)

Tencel Nonwoven samples (Figure 85) were also compared concerning the influence of the fiber diameter. In this case the reverse trend has been observed, the fiber length decreased with increasing fiber diameter. The difference was quite low and in the magnitude of the error.

Hence it is not really possible to conclude how an increasing fiber diameter influences the grinding result. It is also possible that the very small differences concerning the fiber diameter of the examined Tencel fibers do not exhibit any really apparent effects.

Based on this data, it is possible to conclude that an increase of both the fiber length and fiber diameter causes a larger average fiber length. The quantification of the influence of each parameter alone was not possible since adequate samples have not been available.

The reason for the observed phenomena could be explained as following: The probability of cutting a fiber increases with increasing fiber diameter. The fiber stiffness also increases with increasing fiber diameter and thus the chance for passing the sieve increases. Based on these two facts both assumptions, an increase and a decrease of the obtained fiber length, could be conceivable.

For giving a reliable coherence if and how the fiber diameter affects the obtained fiber length more experiments with fiber samples, which only differ from the fiber diameter have to be carried out.

5.1.2 Influence of Additives

a.) Pigments

Relating to the influence of pigments on the obtained fiber length a more suitable relationship could be found. Figure 86 illustrates the comparison of three fiber samples for textile use (Bright with a fiber titer of 1.5 dtex and a starting length of 39 mm), containing different pigments.

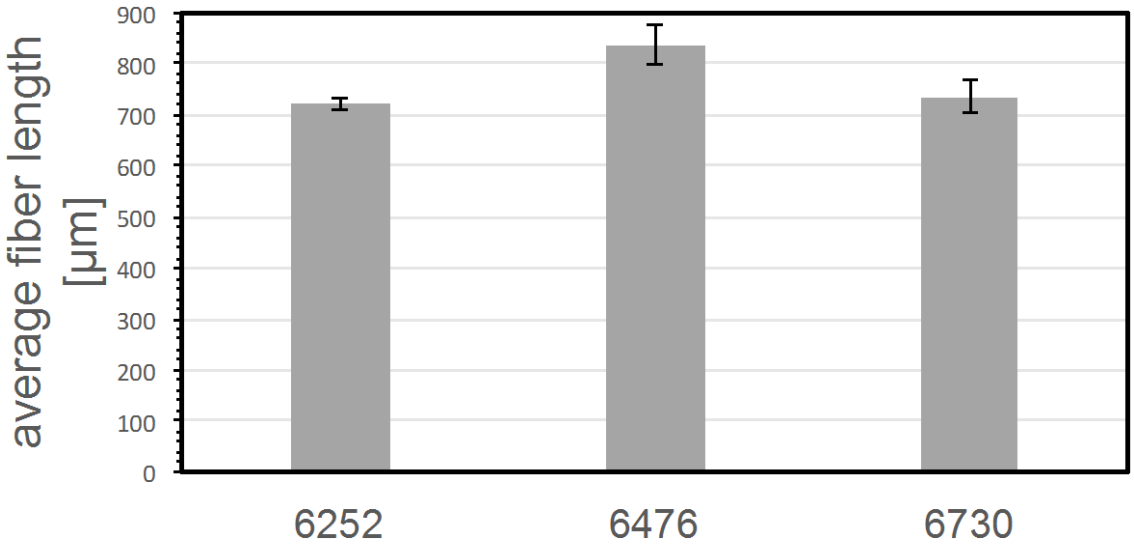

Figure 86: Three different fiber samples for textile use (Bright) containing different types of pigment

Due to the results presented in Figure 86, it is possible to conclude that pigments have an impact on the resulting fiber length. Pigments 6252 and 6730 give a shorter average fiber length than pigment 6476. Pigments represent a discontinuity in the fiber and thus cause lower values for strength. This effect depends on the structure, particle size and density of the pigments. Based on this data it is possible to conclude that pigments have an impact on the grinding result.

b.) Titanium Dioxide

It was also evident that TiO_2, which is used for producing a satin finish, affects the resulting fiber length after the grinding process. Figure 87 compares six fiber samples of which three (Dull) contained TiO_2 and the others (Bright) did not.

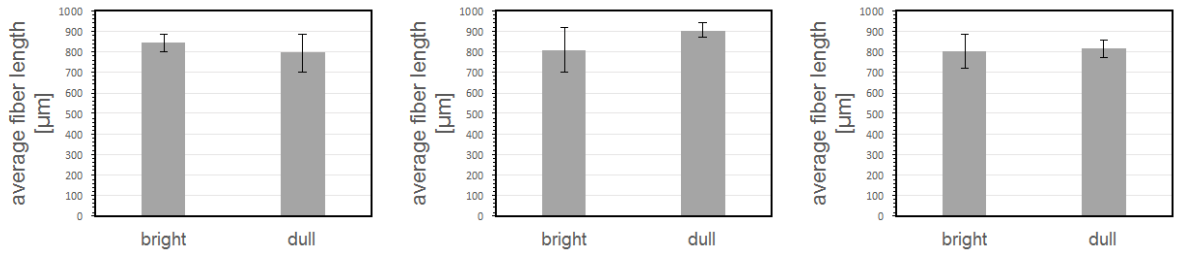

Figure 87: Comparison of fibers with and without titanium dioxide (from left: Tencel, Modal, Viscose)

Titanium dioxide is added directly into the spinning solution before the spinning process. However, titanium dioxide particles cause, during the spinning process when the fiber coagulates, defects. These defects influence the structure and thus the tenacity of the fiber. As a matter of fact the average fiber length of Dull cellulosic fibers is lower compared to pure cellulose fibers. As shown in Figure 87 this effect is apparent for both, Tencel and Viscose fibers. For Modal fibers it is quite difficult to make a statement, hence more experiments will have to be conducted in this field. It could also be possible that another, in this case unknown parameter of the Dull fiber sample, was changed and due to this fact the samples are not comparable. An outliner also could be present.

5.1.3 Influence of Manufacturing Process

Cellulosic fibers can be produced according to different manufacturing processes. In this work three fiber types according to the Viscose, the Modal and the Lyocell process have been investigated. Figure 88 and Figure 89 show the fiber lengths of fibers, which show identical fiber parameters but were manufactured according to different processes.

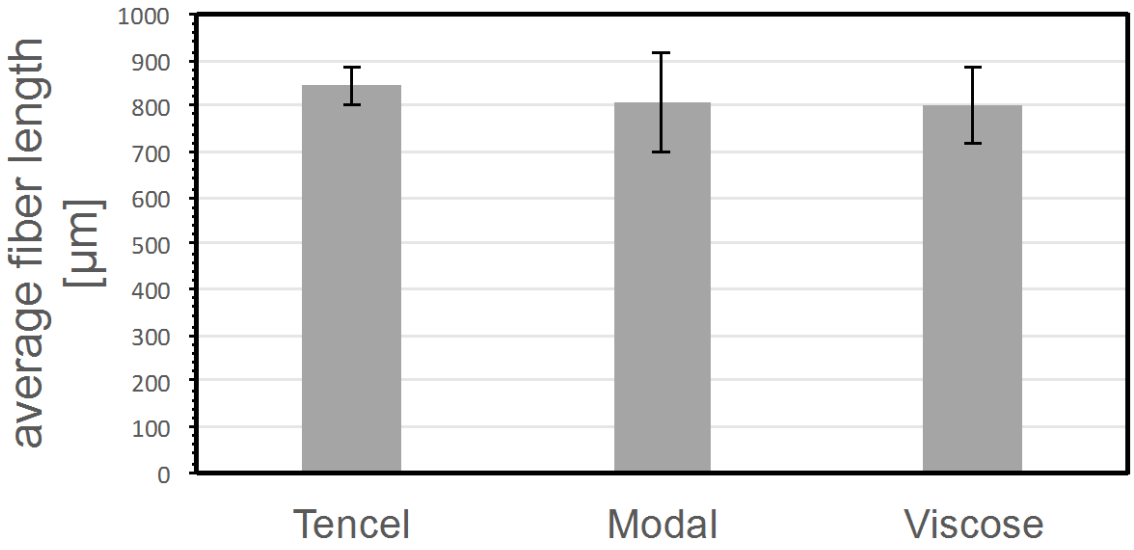

Figure 88: Influence of manufacturing process, Bright cellulosic fibers

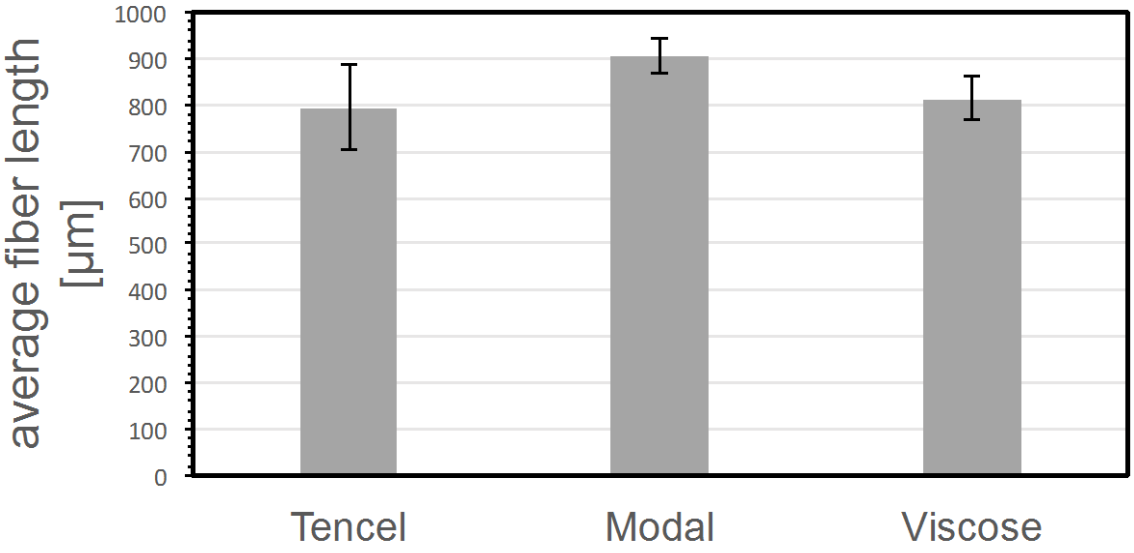

Figure 89: Influence of manufacturing process, Dull cellulosic fibers

Based on Figure 88 and Figure 89 it is possible to conclude that the manufacturing process has an influence on the resulting average fiber length, thus it seems that the fiber tenacity is the relevant parameter.

Table 12 compares the fiber tenacity to the average fiber length.

Table 12: Average fiber length and tenacity of Bright and Dull cellulosic fibers

Manufacturing process	Average fiber length [µm] Bright/Dull	Tenacity [cN/tex] Bright/Dull
Viscose	804/814	25/24 [40]
Modal	809/906	35 [41]/34 [42]
Tencel	845/795	36 [43]/no data

It seems evident that a fiber sample that shows a higher tenacity requires more force to be ground. As a consequence the length of ground fibers should increase with increasing tenacity. However no clear trend could be observed. Further experiments would be necessary to gain more information.

5.1.4 Influence of Process Parameters

It was also investigated how the average fiber length is dependent on the size and shape of sieve meshes as well as the influence of repeated grinding.

5.1.4.1 Sieves

Figure 90 and Figure 91 show the relationship between the obtained average fiber length and the used sieve for sieves of circular mesh and CONIDUR sieves.

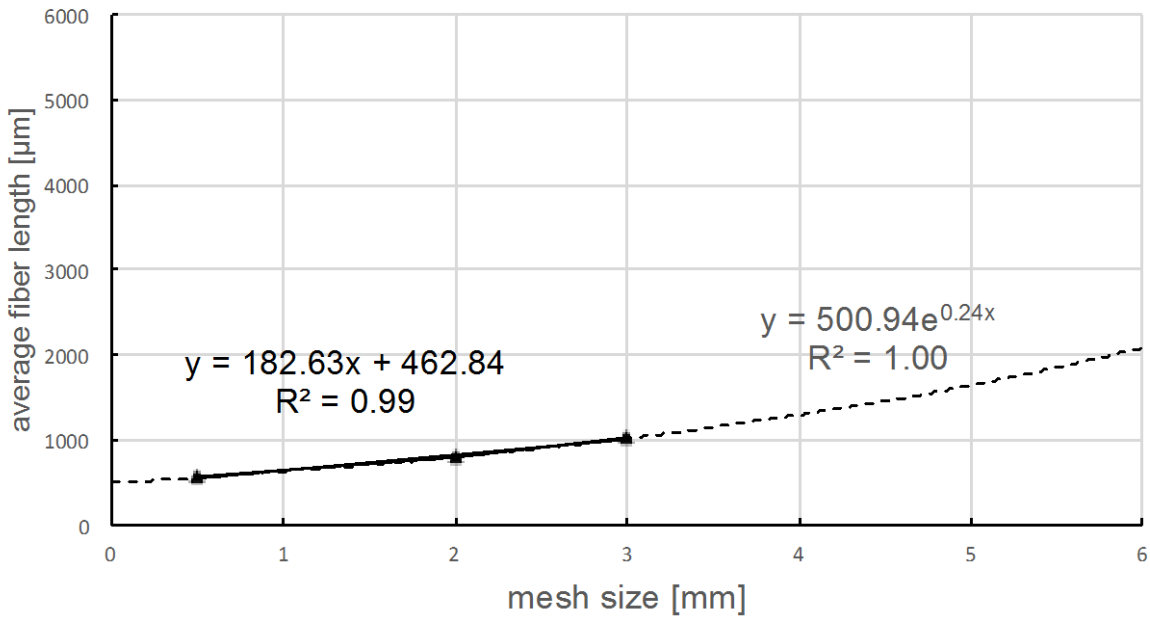

Figure 90: Context between mesh size and obtained average fiber length (for fiber sample Tencel 1.7 dtex 38 Nonwoven) using a sieve of circular mesh

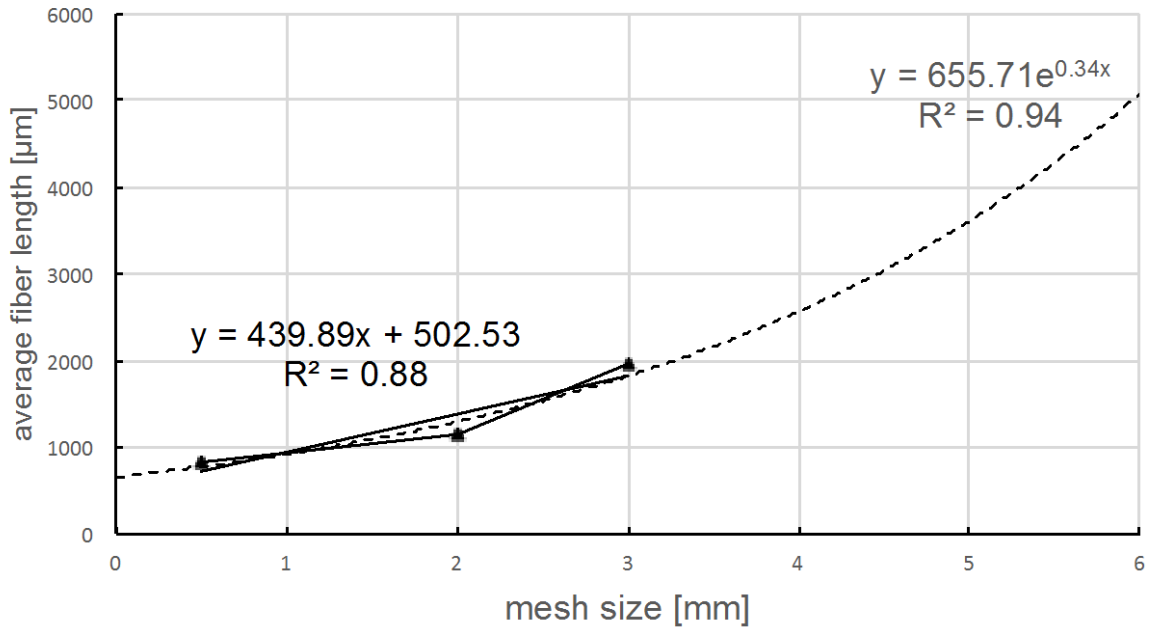

Figure 91: Context between mesh size and obtained average fiber length (for fiber sample Tencel 1.7 dtex 38 Nonwoven) using a CONIDUR sieve

Based on these two Figures it is possible to conclude that using a CONIDUR sieve of the same mesh size results in a larger average fiber length. Due to the shape of the CONIDUR holes the probability for passing the sieve is higher, thus the residence time is reduced. It is obvious that a shorter dwell time will cause less number of impacts of the knives and therefore an increase in obtained fiber length.

$$l = 500.94 * e^{0.24*m}$$

l ... ... average fiber length [µm]

m ... ... mesh size [mm]

Equation 5: Average fiber length as a function of mesh size (sieve of circular mesh)

$$l = 655.71 * e^{0.34*m}$$

l ... ... average fiber length [µm]

m ... ... mesh size [mm]

Equation 6: Average fiber length as a function of mesh size (CONIDUR sieve)

It was tried to fit both, a linear and an exponential function into the experimental data. In both cases an exponential function showed the higher R^2 and therefore the better fitting. Equation 5 and Equation 6 are possibilities to give a forecast which average fiber length will be produced when using a CONIDUR sieve or a sieve of circular mesh with a specified mesh size. These equations are only valid for the fiber sample Tencel with fiber titer 1.7 dtex and 38 mm starting length. For other fiber samples this context must be determined by further tests. Based on these results it can be supposed that there is an exponential relationship between average fiber length and the mesh size of the used sieve. Knowing that there is a mathematical relationship between these two parameters makes it much easier to give a forecast which sieve has to be used to generate a specific average fiber length. A few experiments are enough to define the parameters. Based on this mathematical function it would be quite easy to design an industrial process and define the required mesh size.

5.1.4.2 Process Modes

Concerning process modes two different multi-stage processes have been tested (fiber sample Viscose 1.3 dtex 39 mm starting length Bright). In the first one, a fiber sample was ground using the same 2 mm sieve of circular mesh four times in a row. In the second series the diameter of the used sieve of circular mesh was successively reduced step by step from 3 mm, 2 mm to 0.5 mm. Again it was tried to find a relationship between the average fiber length and the number of grinding steps respectively the number of grinding steps and mesh size diameter.

Figure 92 shows the consequences of the carried out experiments with the 2 mm sieves.

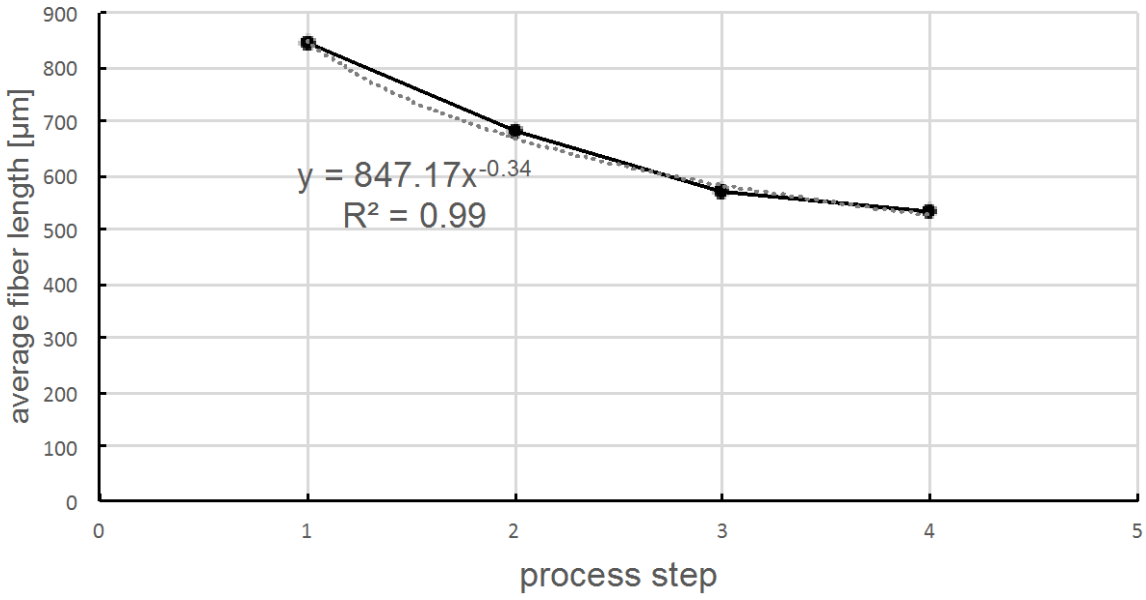

Figure 92: Average fiber length as a function of step number (2 mm sieve of circular mesh)

It could be shown that the relationship between process steps and average fiber length can be described via a power function (Equation 7).

$$l = 847.17 * p^{-0.34}$$

l ... ... average fiber length [µm]

p ... ... process step [−]

Equation 7: Relationship between process step and average fiber length

Again, this equation is only valid for the mentioned fiber sample (Viscose 1.3 dtex39 mm starting length Bright). For another sample the factors have to be investigated in a test. The experiments showed that with this repeated use of a sieve it is possible to produce fibers, which are much shorter than the nominal mesh size of the sieve. The length reduction per step is getting smaller with every step and obviously goes towards zero. There might be no further length reduction of the fiber sample as the length is small enough to pass the sieve without any impact of the knives.

In the second experimental series the mesh size of the used sieve of circular mesh was reduced step by step from 3 mm, over 2 mm to 0.5 mm. Figure 93 shows the results of experiments.

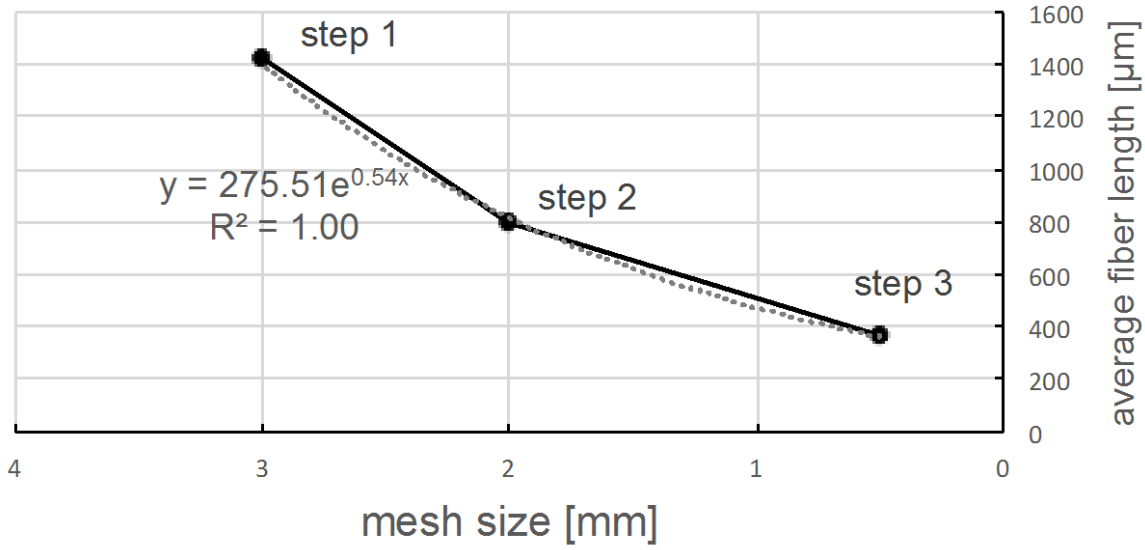

Figure 93: Repeated grinding with lowering the mesh size

Equation 8 describes the relationship between average fiber length, grinding step and mesh size.

$$l = 275.51 * e^{0.54*m}$$

l ... ... average fiber length [µm]

m ... ... mesh size [mm]

Equation 8: Relationship between mesh size and average fiber length in case of lowering the mesh size every step

It could be shown that if the mesh size is reduced with every step like shown in Figure 94, the resulting average fiber length follows an exponential function.

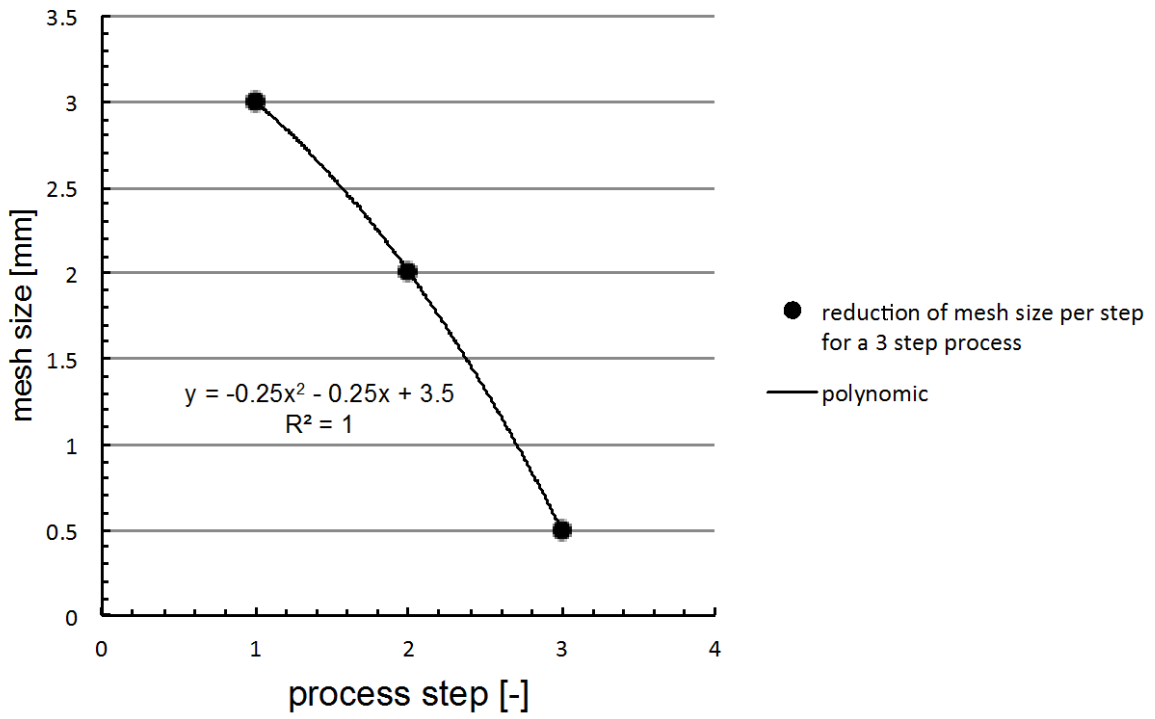

Figure 94: Relationship between mesh size reduction and process step

With this kind of multi-step grinding process it is possible to produce a much shorter average fiber length than with the first method. Again the phenomenon that the length reduction becomes smaller with every step is obvious. However, a problem that appeared in this experimental series was that when using the 0.5 mm sieve a lot of fiber material stayed in the mill due to a kind of fleece that was formed on the sieve. As a result the received, ground fiber amount was reduced a lot and finally the mill had to be opened and cleaned. Thus an industrial process would not be viable. This phenomenon may occur due to electrostatic charges.

5.1.4.3 Influence of the Airflow

As last process parameter influencing the fiber length the effect of the airflow was investigated. A side channel blower was used and a shutoff damper controlled the flow. Figure 95 shows the context between shutoff damper position and the airflow (empty conduit).

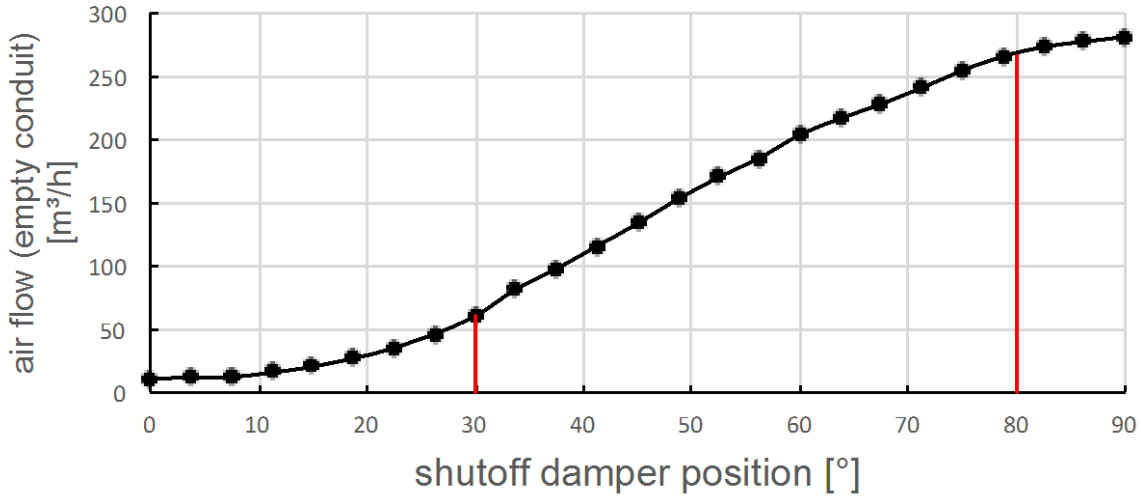

Figure 95: Relationship shutoff damper position/airflow

As shown in Figure 95, there is a linear relationship between the position of the shutoff damper and the airflow at angles between 30° and 80°. Fiber samples were taken directly after the cutting mill using a sample probe at two different shutoff damper positions to find out how the airflow influences the average fiber length.

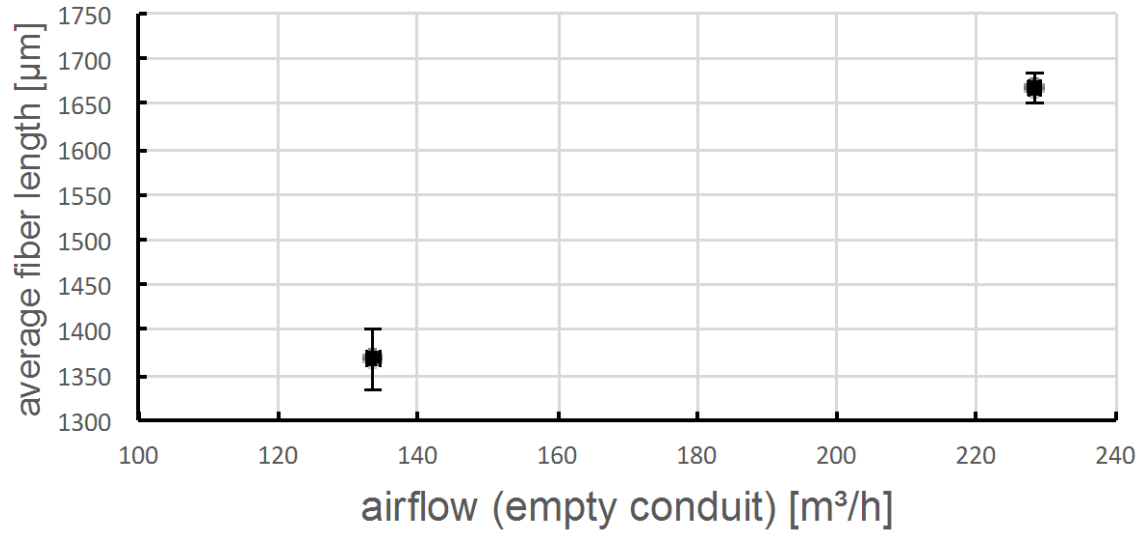

Figure 96: Relationship between airflow and average fiber length

Figure 96 shows that the average fiber length increases with rising airflow. A higher flow rate implicates a shorter dwell time of the fiber in the mill. The probability for cutting is therefore smaller. The other way round implicates a lower flow rate a higher dwell time and therefore the probability for cutting is higher. Due to the fact that only

two measurements were carried out, it is not possible to say that the relationship between the two parameters is linear but since the volumetric flow rate increases linear in this area (as shown in Figure 95) it could be so. For detailed information more experiments would be necessary.

5.2 Bulk Density as a Characteristic for Fiber Grinding

Determining the bulk density of a fiber collective is an absolutely simple process. Due to the fact that no expensive equipment and no special trained staff are necessary it seems to be a quite economic tool for process control in the fiber industry. After taking a sample from the process, a defined volume of this sample has to be weighted and the density can be calculated easily. So it appears to be a quite suitable tool for at line process characterization and analysis. However, the applicability is limited.

An applicable field of bulk density for characterizing a grinding process might be situations where a process parameter but not a fiber parameter is changed.

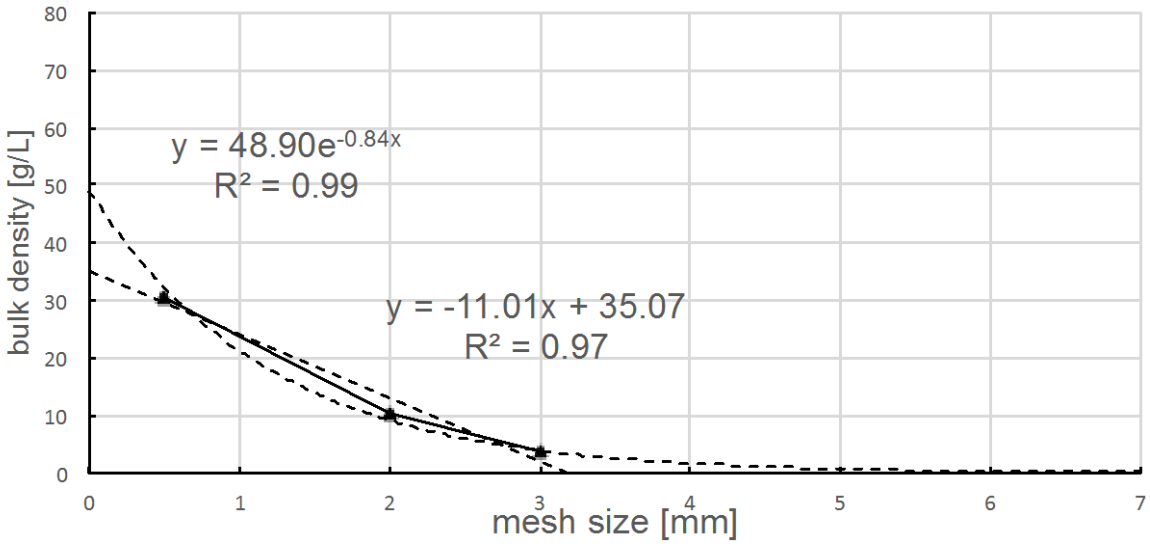

Figure 97: Bulk density vs mesh size when using CONIDUR sieves (fiber sample: Tencel 1.7 dtex 38 mm starting length Nonwoven)

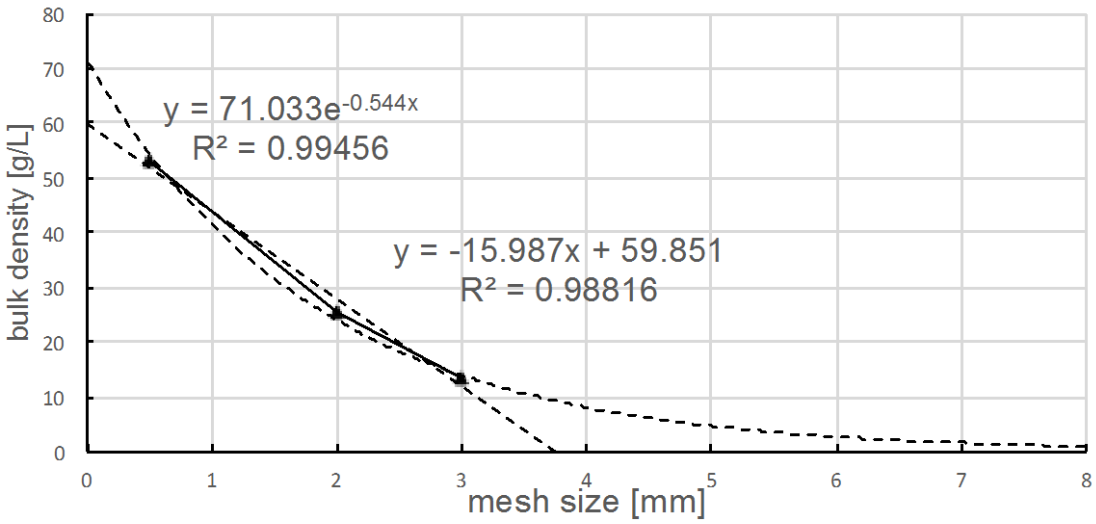

Figure 98: Bulk density vs mesh size when using sieves of circular mesh (fiber sample: Tencel 1.7 dtex 38 mm starting length Nonwoven)

Figure 97 and Figure 98 show the relationship between the mesh size and the resulting bulk density.

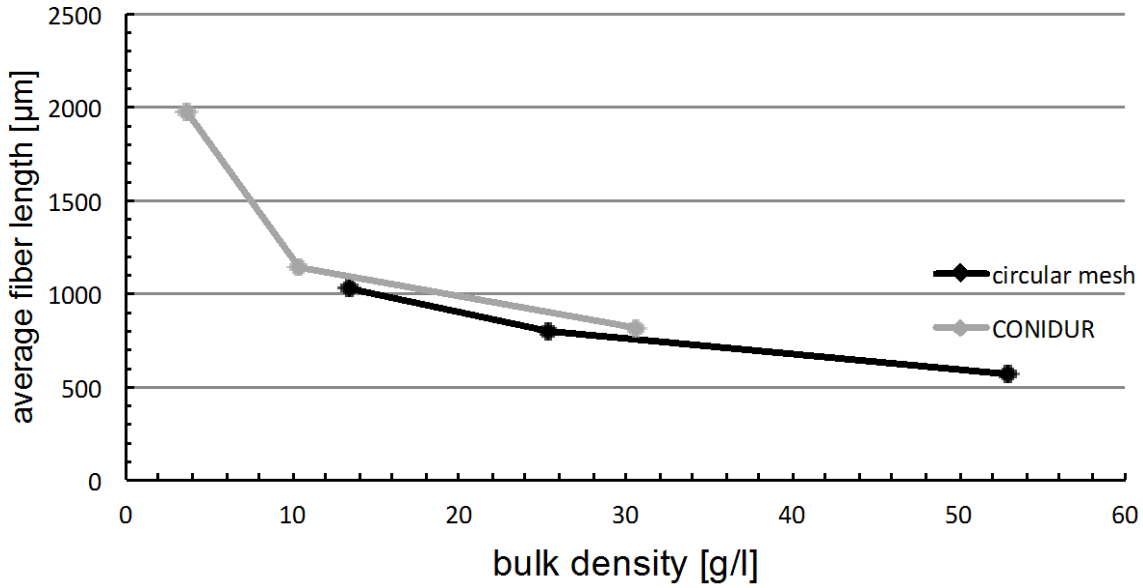

Figure 99: Relationship between bulk density and average fiber length (fiber sample: Tencel 1.7 dtex 38 mm starting length Nonwoven)

Figure 99 shows the relationship between the produced fiber length and the bulk density (combination of Figures 90, 91, 97 and 98). This relationship is only valid if always fibers with the same characteristics like material, fiber titer etc. are ground. Then it can be used as an easy and cheap quality check for industry. It is a fast

alternative for determining approximate values, but determining the bulk density cannot replace a detailed fiber length analysis.

Figure 100 shows an application for which this analysis tool is absolutely not usable.

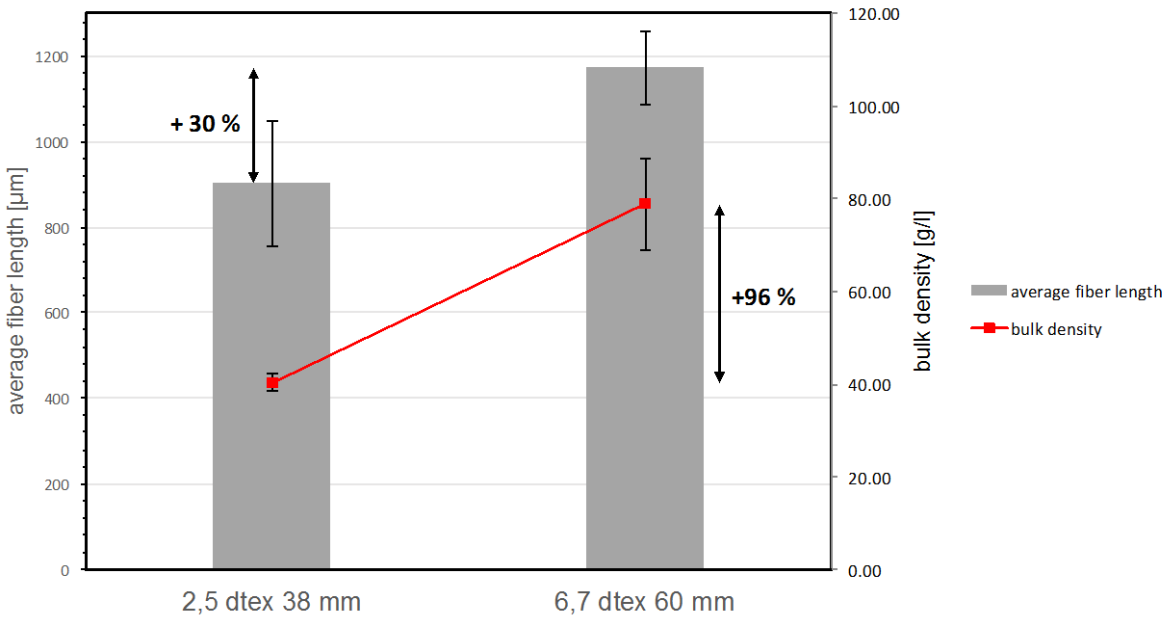

Figure 100: Bulk density and average fiber length of two Tencel samples containing ZnO and with different fiber parameters

In this case two density relevant fiber parameters differ. Hence determining the bulk density to characterize the grindability is senseless.

6 Conclusion and Outlook

a.) Fiber Grinding

In the field of fiber grinding it was possible to find a first systematic approach how different fiber and process parameters influence the grinding process. It was possible to show that additives like pigments or TiO_2 have an influence on the grinding process. The process of fiber manufacturing also has an influence on the resulting fiber length. It was not possible to quantify the influence of the fiber titer. It was possible to show that a combination of increasing starting length and titer leads to an increase of the average fiber length. Relationships between fiber length and sieve were found concerning sieve types and mesh size and mathematic correlations were set up. Multi-stage grinding processes with the same and decreasing mesh size were analyzed and correlations for calculating the processes were set up.

Concerning the fiber grinding process further investigations are required to evaluate more parameters and their influence on the fiber grinding process. The aim of the future work will be the development of a mathematical model for describing the fiber grinding process in a cutting mill.

b.) Fiber Characterization

In the field of fiber characterization a few new insights were gained.

It was shown that the fiber analyzer MorFi gives precise and repeatable results, but is a quite expensive characterization tool. Using a microscopy system is a reliable method for characterizing fibers but it is very elaborate and also expensive. Furthermore it was shown that a microscopy system is limited to dry dispersed samples. Using a scanner instead of a microscopy system makes it possible to use wet dispersion. Coloring the fibers using a reactive dye can easily solve problems with the contrast between white fibers and the background when using a commercially available scanner. The new developed fiber length determination technique seems to be suitable and represents a cheap alternative to commonly used fiber characterization tools.

Relating to the new characterization method for fiber length determination a few further improvements have to be made. The focus should be on the development of an automated procedure to exclude crossed fibers and fiber networks from the analysis. This process might be based on particle parameters, which are commonly used in particle characterization software. With the introduction of the new, 64bit ready, version of Scientific Counter it will be possible to analyze larger images. Concerning the contrast problems a more sophisticated scanner could bring improvement.

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