Effect of alkali pre-treatment on Trichoderma cellulase treatments of cellulose fibers

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ABSTRACT

In modern textile processing, environmentally friendly enzyme treatments are more and more replacing techniques that use concentrated chemicals and aggressive co-additives. Cellulase hydrolysis of cellulosic fabrics improves handle and drape, removes pills and gives color brilliance, and in denim finishing cellulases were introduced to replace pumice stones for creating the stone-wash effect. But until now no treatments using intensive cellulose degradation could be established.

In this thesis, viscose fabrics were pre-treated with liquid ammonia and NaOH solutions. The pre-treatment was varied in alkali concentration, time and drying conditions. Subsequently, the samples were subjected to cellulase hydrolysis. Microscope and SEM pictures were taken; weight loss, reducing sugar and protein content in solution, water retention value and tensile strength were determined.

It was found, that the activity of cellulases is increased for viscose and that short time alkali pre-treatment reduces tensile strength due to changes in the substrate. The drying and drying conditions (wet, line dry and freeze dry) have great impact on the hydrolysis rate. A connection between velocity of protein loss in solution and changed water retention value was established. Weight loss of 80% of cellulose fabric was achieved within 4 hours of enzyme hydrolysis.

This effect was also introduced into textile garment finishing. Local application by printing alkali-containing pastes permits selective surface modification. Indigo ring-dyed denim fabric can be used as an example. The intensified local degradation of the fibres on the surface of the fabric leads to higher contrast in the wash-down.

Key words:

Alkali, Cellulase, Cellulosic material, Cellulose activation, Denim, Hydrolysis, Pre-treatment, Protein concentration, SEM, Tensile strength, Water retention value.

ZUSAMMENFASSUNG

In der modernen Textilproduktion werden Verfahren mit konzentrierten und aggressiven Chemikalien mehr und mehr von umweltfreundlichen Enzymbehandlungen abgelöst. Enzymatische Hydrolyse von zellulosischen Geweben bewirkt einen verbesserten Griff, und es kommt zu einer Verbesserung der Pilleigenschaften und Erhöhung der Farbrillanz. Weiters werden Cellulasen bei der Denimherstellung eingesetzt, wo sie den Einsatz von Bimsstein ersetzen. Bis jetzt wurden aber noch keine großtechnischen Verfahren entwickelt, bei denen eine tiefgreifende Faserzerstörung mit Hilfe von hydrolytischen Enzymen erwünscht und möglich ist.

Im Zuge dieser Diplomarbeit wurden Viskosegewebe mit flüssigem Ammoniak und Natronlauge vorbehandelt. Dabei wurden die Konzentration an Lauge, die Behandlungszeit und die Trocknungsbedingungen variiert und das Gewebe anschließend einer enzymatischen Hydrolyse mit einer Enzymmischung aus Cellulasen unterzogen. Die Veränderungen im Gewebe wurden mittels mikroskopischen und elektronenmikroskopischen Aufnahmen, Messung des Gewichtsverlustes, Analyse der freigesetzten reduzierenden Zuckern und der Veränderung des Proteingehaltes während der Enzymbehandlung sowie der Veränderung der Zugfestigkeit untersucht.

Es wurde festgestellt, dass kurzzeitige alkalische Vorbehandlungen zu Einbußen in der Zugfestigkeit führen. Die Aktivität der Cellulasen war erhöht und stark abhängig von der Trocknung und den Bedingungen nach der alkalischen Vorbehandlung (Nass, Lufttrocknung und Gefriertrocknung).

Außerdem konnte eine Verbindung zwischen dem Proteinverlust in der Behandlungslösung und der Änderung im Wasserrückhaltevermögen des Gewebes beschrieben werden. Nach vier Stunden enzymatischer Behandlung wurde ein Gewichtsverlust von 80% erzielt.

Dieser Effekt wurde bei einem Einsatz des Verfahrens beim Finishing von Kleidungsstücken bestätigt. Indigo-ringgefärbtes Gewebe diente dabei als Demonstrationsobjekt. Dabei wurde eine alkalische Druckpaste oberflächlich aufgebracht. Nach der enzymatischen Hydrolyse konnte ein hoher Kontrast zwischen behandelten und unbehandelten Gewebeteilen erzielt werden.

Schlagworte:

Cellulase, Lauge, Denim, Hydrolyse, Proteinkonzentration, SEM, Vorbehandlung, Wasserrückhaltevermögen, Zelluloseaktivierung, Zellulosegewebe, Zugfestigkeit.

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1. INTRODUCTION

Environmentally friendly enzyme treatments are more and more replacing techniques that use concentrated chemicals and aggressive co-additives in textile processing. Amylases have been used for desizing for more than 100 years, pectinases may replace alkali at the scouring process, and cellulases have been in use since more than ten years ago (Guebitz and Cavaco-Paulo 2001). Cellulase hydrolysis of cellulosic fabrics improves handle and drape (e.g. linen), removes pills (defibrillation) in bio-polishing of man-made cellulose fabrics, gives color brilliance, increased luster and color protection and brightness (removal of graying) in textile washing (Klahorst et al. 1994; Kumar et al. 1994; Kumar and Harnden 1999; Miettinen-Oinonen 2001). In denim finishing, cellulases were introduced to replace pumice stones for creating the stone-wash effect where the indigo ring-dyed fabric is garment washed in numerous techniques to obtain certain variations in colour depth, shades and contrast (Cavaco-Paulo et al. 1998; Etters 1992; Heikinheimo et al. 2000).

Enzymatic treatments cause changes in mechanical properties of substrates and may also lead to unique textile effects or ecological improvements in existing procedures (Bredereck 1995; Kumar and Harnden 1999; Lee et al. 2000). In general, controlled enzymatic attack results in moderate weight and strength losses of cellulose fabrics (Klahorst et al. 1994; Cavaco-Paulo and Almeida 1996; Heikinheimo et al. 1998). Control of enzyme activity is essential to avoid high losses in tensile strength. In most cases weight loss is limited to between 2 and 7% (Kumar et al. 1995; Pedersen et al. 1993). Established techniques therefore demand gentle hydrolysis to avoid fabric tendering (Tyndall 1992).

New areas of application were found which require partial or complete hydrolysis of the cellulosic parts of blends. In production of special types of embroideries the pattern is sewn onto a cellulosic fabric with polyester yarn. The cellulosic fabric, which acts as the ground, is then removed by hydrolysis to yield the embroidery structure (Doebel 1999; Klobes et al. 2003; Vasconcelos and Cavaco-Paulo 2006; Schimper et al. 2008).

The cellulolytic system required for this process should be an aggressive mixture to perform total hydrolysis in a short time. A total culture acid cellulase preparation (TC) which derives from the filamentous fungus *Trichoderma reesei* will fit this requirement.

In general, enzymatic hydrolysis by cellulase is a result of different actions. Several authors have reviewed the characteristic mode of action and behavior of cellulases (Enari et al. 1987; Cavaco-Paulo 1998; Focher et al. 1991; Heikinheimo 2002; Montenecourt 1983; Pere et al. 2001; Schimper et al. 2004; Väljamäe 2002; Wood 1991). Three principal types of enzymes work synergistically to degrade cellulose into glucose by cleaving the β-1-4-glycosidic bonds of the cellulose chain. Endoglucanases (EG, EC 3.2.1.4) and cellobiohydrolases (CBHs, EC 3.2.1.91) degrade the cellulose chain primarily into cellobiose. β-Glucosidase then completes the hydrolysis of cellobiose to glucose (Azevedo et al. 2001).

The fungus *T. reesei* produces at least four endoglucanases: EG I, EG II, EG III and EG V in a TC preparation. EG I and EG II are the most abundant endoglucanases, each accounting for 5 – 10% weight of the whole cellulase preparation (Väljamäe 2002). Endoglucanases act randomly at the most accessible points of the cellulosic chain and create new chain ends for exoglucanase attack. Cellobiose is not attacked

by endoglucanases. It is a strong inhibitor for EG and CBHs, which are the main producers of cellobiose.

Cellobiohydrolase is the most abundant protein in total culture filtrates of *T. reesei* and includes mainly two types of exoglucanases: CBH I and CBH II. CBH I attacks at the reducing end of the cellulose chain and comprises about 60% of the total cellulolytic proteins. The main product of hydrolysis is cellobiose and cellotriose. CBH II, the second exoglucanase, comprises about 20% of the total produced and acts from the non-reducing end of the chain. As for CBH I, the main product is cellobiose. At least one β-glucosidase is produced by *T. reesei,* and it completes the hydrolysis of the cellulose by cleaving cellobiose to glucose. It is now widely accepted that endoglucanases and cellobiohydrolases act synergistically. Hence, a TC cellulase preparation was used in this study to achieve fast degradation.

Viscose, a man-made fiber, is reported to be easily degradable by cellulases compared to other man-made fibers (Carillo et al. 2003; Schimper et al. 2004). Alkali treatment, such as mercerization in textile industry, improves cellulase attack due to a homogeneous and opened fiber structure (Buschle-Diller and Zeronian 1994; vanWyk 1997).

Rath (1972) reported that mercerizing of viscose resulted in improved alkali solubility compared to cotton. Usually mercerization is performed using sodium hydroxide solutions with alkali concentrations between 27 and 33°Be (206 – 266 g/L NaOH). When removing the caustic soda from the fiber by rinsing with water, sodium hydroxide concentrations until 0% are continuously passed through. During the rinsing process, the range between 8 – 10% is passed where man-made fibers show maximum solublility (Figure 1). This effect is strongly dependent on temperature, hence cellulose

solubility in caustic soda is decreased to a minimum at temperatures of 60 – 70°C. For gentle mercerizing processes it is recommended to avoid processing at room temperature.

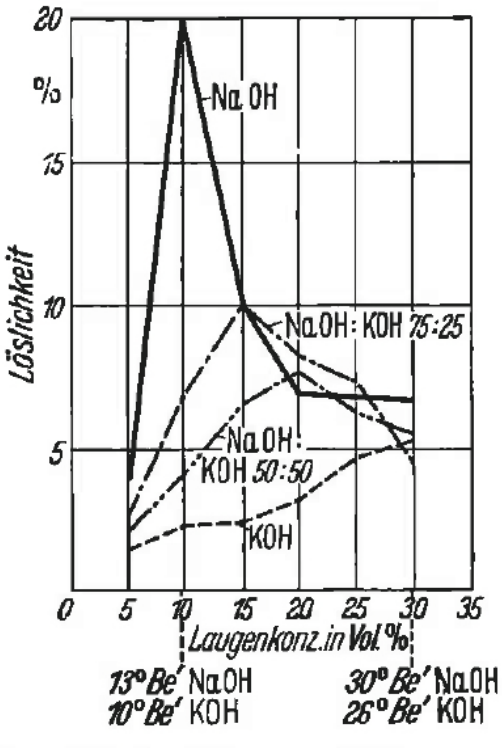


Figure 1. Solubility of man-made fibers in NaOH, KOH and mixtures thereof depending on the concentration at 20°C (Rath 1972).

Few studies have reported treatments to enhance degradation rates of cellulases in textile area. In this investigation the effect of preceding caustic soda treatment on observed hydrolytic activity of *Trichoderma* cellulase treatments towards regenerated cellulose will be reported. The impact of different alkali concentrations and treatment time as well as drying and drying conditions on fiber structure was investigated and will be discussed.

Additionally, local activation of the cellulose surface for cellulase degradation was studied on denim samples for visualisation of the effect. The punctual activation was done by application of concentrated alkali pastes on the fabric surface. Followed by the activation step, the fabric was washed and neutralized and subsequently cellulase treated. Activation tests with activated indigo ring-dyed samples are shown to visualize the effects obtained.

2. MATERIALS AND METHODS

2.1 Materials

A viscose (CV) fabric made of 38 mm staple fibers (1.3 dtex) was used as the substrate. Plain woven samples of fabric (yarns of 20 tex in warp, 24 tex in weft direction, 19 twist per inch in both warp and weft directions and weight of 143 g/m^2) with 29/37 picks/ends were obtained from Lenzing AG, Austria.

For local activation experiments indigo-dyed denim fabrics, supplied by DyStar (Frankfurt a.M., Germany) were used. The desized twill woven cotton samples were called Denim I (438 g/m^2 with 19/27 picks/ends) and Denim II (466 g/m^2 with 18 picks/ends).

The cellulase enzyme used in these experiments (provided by Genencor International, USA) was applied without further purification. The nominal activity of the total culture filtrate was 3.5 IU/mg. In tests performed at our laboratory, we obtained the following values: 91 filter paper units/g cellulase-activity and 3100 units/g carboxymethylcellulose-activity according to the method described by Ghose (1987), and 7.4 IU/g activity on Avicel according to the method described by Wood and Mahlingeshwara (1988).

A stock solution of 50% (w/w) caustic soda technical grade (K. Deuring GmbH & Co, Austria) was diluted with deionized water to concentrations of 1.0, 2.2, 3.5 and 4.9 mol/L NaOH for sample pre-treatment.

The printing paste for surface activation experiments contained of 73% w/w alkali stable thickener (20 g/L Prisglon GT 1000 M, Bezema AG, Swiss) and 27% w/w NaOH 50%.

All chemicals used for analysis were analytical grade and were kindly provided by Sigma Aldrich Production GmbH, Switzerland.

2.2 Treatment methods

Impact of different alkali concentration and treatment time.

Viscose fabrics were pre-treated in one step which consists of immersing in alkali solutions for one or two minutes at a liquor ratio of 1:3 (w/v). No tension was applied to the fabrics. A range of concentrations (0 – 4.9 mol/L) was used in the impregnation solution. After squeezing in a padder, samples were rinsed two times with deionized water and then neutralized in a solution of 20 mmol/L acetic acid for five minutes. The alkali uptake was found to be between 100 – 250%, depending on alkali concentration. After neutralization the fabrics were enzyme treated. Therefore the samples were divided into several parts, where the first part of the sample was air dried, the second freeze dried and the third part was kept in wet state until cellulase treatment. Samples without alkali pre-treatment served as control. Liquid ammonia treated samples — the treatments being performed at Martini, Germany —, were used as received and subjected to cellulase hydrolysis.

Cellulase enzyme was diluted with 50 mM sodium acetate buffer pH 4.8 to a concentration of 30 mg protein per mL. Hydrolysis was carried out at 55°C (+/-1°C) and at a liquor ratio of 1:25 (w/v) in a laboratory dyeing unit (Pretema Multicolor Typ MC 360; Caromatic, Switzerland). The treatment solution was pumped through the fabric at controlled agitation level. After 4 hours treatment, enzymes were inactivated at pH 10 (sodium carbonate) and 75°C, rinsed with deionized water, and air dried. Each treatment was done in duplicate. Figure 2 gives a schematic overview of sample treatment and analytical procedures and Table 1 assigns a sample code to the different caustic soda treatments which are described in the text.

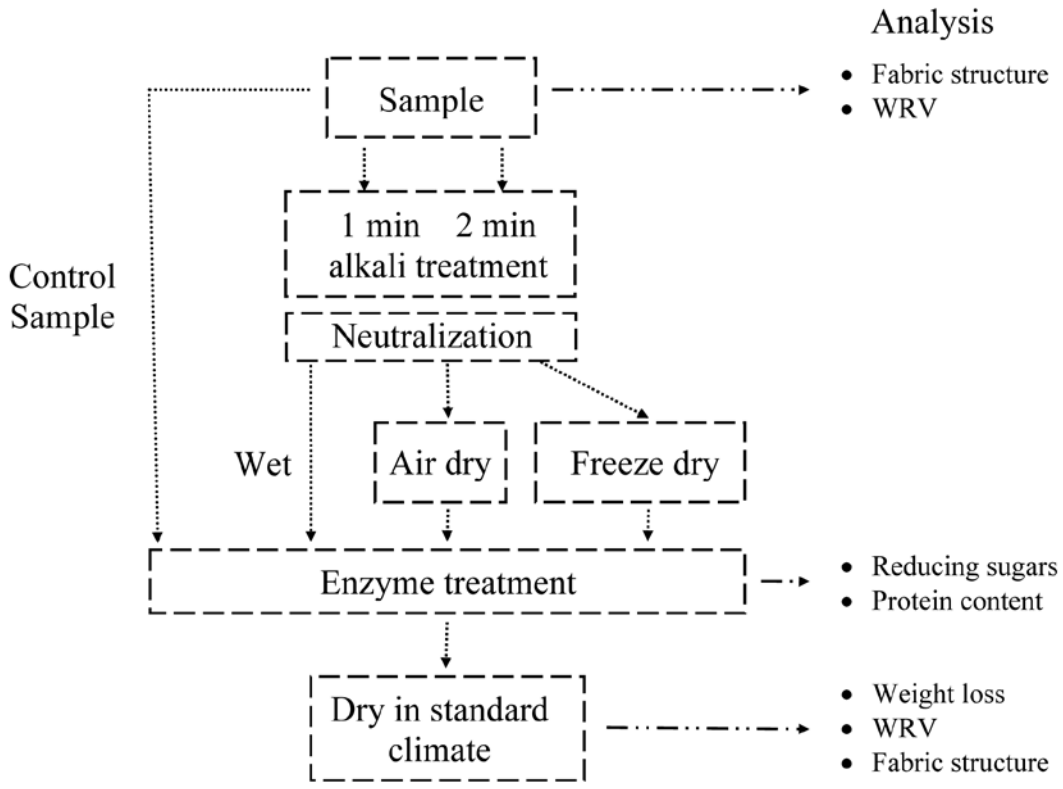


Figure 2. Schematic overview about process structure and analytical procedures of viscose samples.

Table 1. Sample code of the different caustic soda treatments of viscose samples. Numbers 1.0 – 4.9 on the first position indicate the alkali concentration, alkali pre-treatment time is mentioned as the numbers 1 and 2 (one or two minutes) on the second position, and the drying method before enzymatic hydrolysis is described by the small letter on the third position (without drying: w; line (air) drying: d; freeze drying: f).

	NaOH pre-treatments						
Drying	1.0 mol/L		2.2 mol/L		3.5 mol/L		4.9 mol/L
conditions	1 min	2 min	1 min	2 min	1 min	2 min	1 min
Wet	1.0-1-w	1.0-2-w	2.2-1-w	2.2-2-w	3.5-1-w	3.5-2-w	4.9-1-w
Line dry	1.0-1-d	1.0-2-d	2.2-1-d	2.2-2-d	3.5-1-d	3.5-2-d	n. p.
Freeze dry	1.0-1-f	1.0-2-f	2.2-1-f	2.2-2-f	3.5-1-f	3.5-2-f	n. p.

Local activation of the cellulose surface by printing experiments

The alkali activation of the surface was done by a flat screen-printing. Stripes were used as pattern to be printed to demonstrate the effects obtained. After one minute of reaction time the paste was washed off. The samples were washed three times with cold water and once at 40°C with 1% acetic acid solution to neutralize all residual alkali trapped in the fabric.

After neutralisation fabrics were subjected to enzyme hydrolysis as described for viscose samples. The treatment time in the cellulase solution was changed to 45 min.

2.3 Testing

Reducing sugars as glucose: Soluble reducing sugars, calculated as glucose, were determined using the neocuproine method (Cavaco-Paulo et al. 1996) as mg glucose per mL of solution (cGlc) [mg/mL].

Protein in liquor: Total protein in solution was measured by the Bradford method using Coomassie Brilliant Blue G-250 as acid dye for proteins and bovine serum albumin as standard (Bradford 1976). The protein content was indicated as mg protein per mL solution (cP) [mg/mL].

Water retention value: The quantity of water fibers can absorb under strictly controlled conditions is expressed by the water retention value (WRV). The value is calculated according to Okubayashi et al. 2004, as the ratio between the mass of water retained in the fibers after 2 hours of soaking and 20 minutes centrifugation, and the mass of dry sample (105°C, 4 hours). All samples measurements were repeated four times.

Fabric weight loss: The weight loss of fabrics (WL) was calculated as the percentage difference in weight between untreated (control) and enzyme or enzyme and alkali treated fabric.

Tensile strength: The breaking strength in fill direction was tested on an Instron Tensile Tester (Model H1343, Instron AG, Switzerland) according to the reveled strip test (ISO 13934-1:1999) with 200 cN pre-tension at a gauge length of 10 cm and a rate of extension of 5 cm/min.

Microscopic images: A Krüss Optronic microscope (MSZ 5600) with a magnification of ca. 100x was used.

SEM analysis of fabric and fibers: A Phillips XL30 ESEM-FEG was used to observe the fabrics surfaces and fibers. The magnification powers were 100x, 1000x and 10000x and images were recorded for each magnification.

Lab-values of denim samples: Colour of samples was measured as Lab-values. Lab-values were determined using the Chroma Meter CR-200 data processor and measuring head, Minolta, Germany. The system was calibrated for each set of 5 measurements with the standard white plate CR-A43. The measuring head had a measuring area of 10 mm diameter. Total colour difference (ΔE^*_{ab}) was measured using the L*, a* and b* colour coordinates and defined by Eq. I.

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad \text{(I)}$$

Experimental results of Lab-values were statistically treated using the program SPSS.

Statistical analysis of data was conducted at the 0.05 level of significance. Error bars in graphs represent the 95% confidence intervals.

3. RESULTS

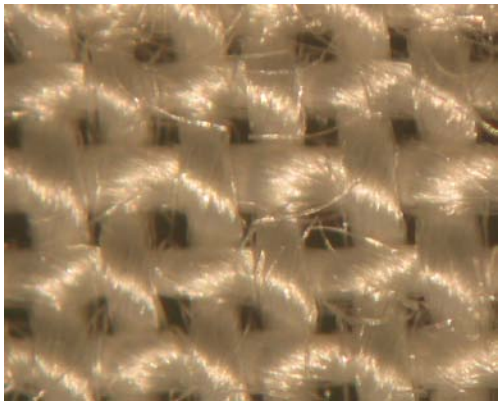
3.1 Impact of different alkali concentrations and treatment times

Buschle-Diller and Zeronian (1994) reported increased weight loss during enzymatic cotton hydrolysis after mercerization. Their slack and tension mercerization was carried out with 5.0 mol/L NaOH at 0°C for one hour.

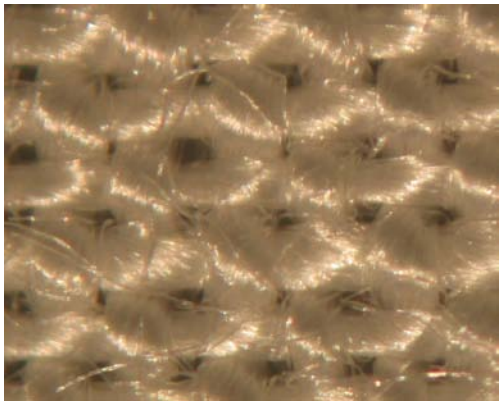
In this study, viscose fabrics were pre-treated for one or two minutes with different concentrations of NaOH without any tension applied, followed by hydrolysis with cellulases.

The pick up of caustic soda solutions increased from ca. 100% at 1.0 mol/L to 200% at 3.5 mol/L and then decreased at higher values. No significant difference in pick up was found between impregnation time of 1 and 2 minutes. The washing was performed at a liquor ratio of 1:60 (w/v) and the pH at the washing steps was between 11 – 12.5 for the first, 8.5 – 10.5 at the second, and between 4.0 and 5.5 at the neutralizing bath.

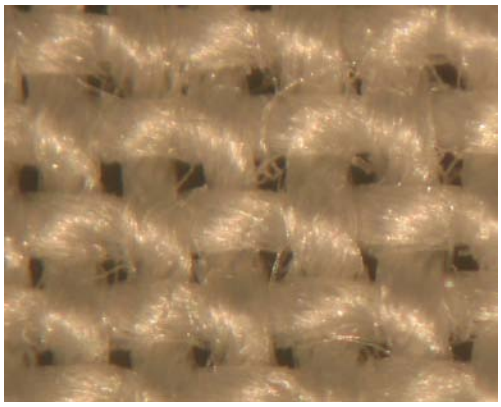
Figure 3 shows photographs of 1 minute alkali-treated viscose samples before enzymatic hydrolysis in a stereo-microscope with a magnification of ca. 1:100. 1.0 and 2.2 mol/L treatments do not clearly show differences from the blank sample. However, the 3.5 mol/L treated sample shows agglutination of fibers, and fiber damage is obvious.



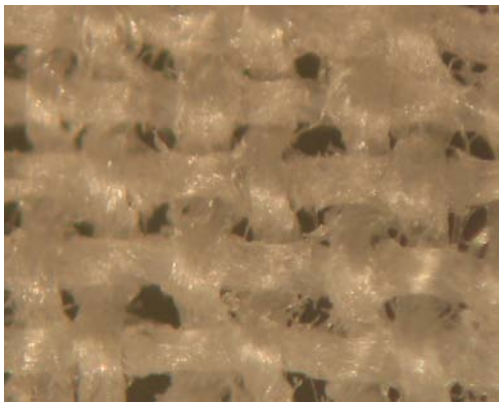
a. Untreated



b. 1 mol/L, 1 minute (1.0-1-d)



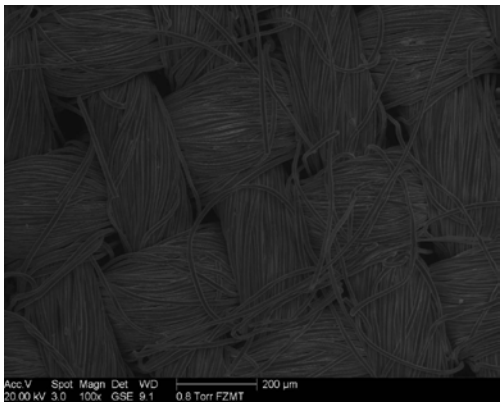
c. 2.2 mol/L, 1 minute (2.2-1-d)



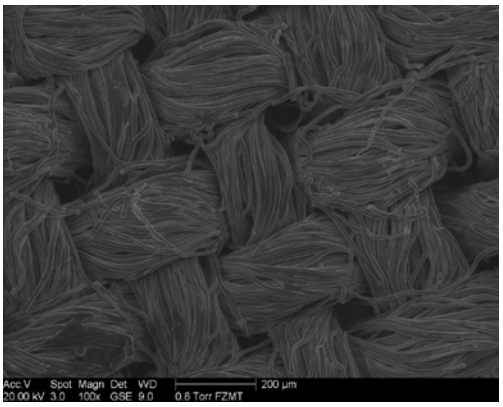
d. 3.5 mol/L, 1 minute (3.5-1-d)

Figure 3. Microscopic images of untreated and alkali-treated viscose samples.

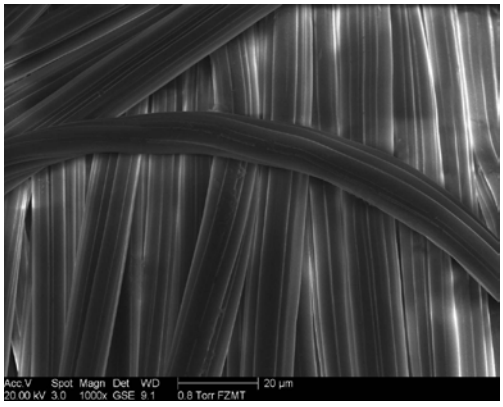
The control sample (Fig. 3a) and the 3.5 mol/L, 1 minute treated sample (Fig. 3d) was visualized in SEM. The samples were examined under the microscope at various places. Representative pictures are shown here. Following magnifications were chosen: 100x to show the fabric structure; 1000x to show the yarn structure and 1000x to show the fiber structure. Figure 4a-c shows the untreated blank sample with an ordered fiber surface (Fig. 4c). Figure 4d-e shows the alkali-treated sample 3.5-1-d with a disordered surface. Fiber damage is obvious in all pictures. The surface appears to have crocks.



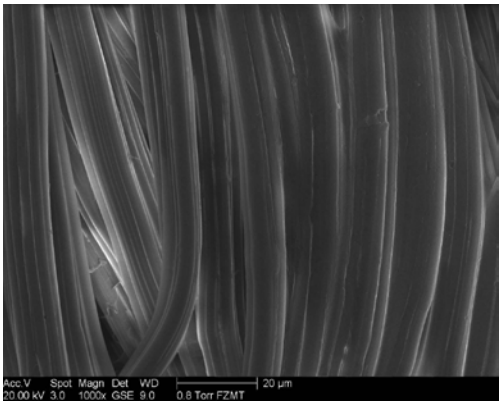
a. Untreated, magnification 100x



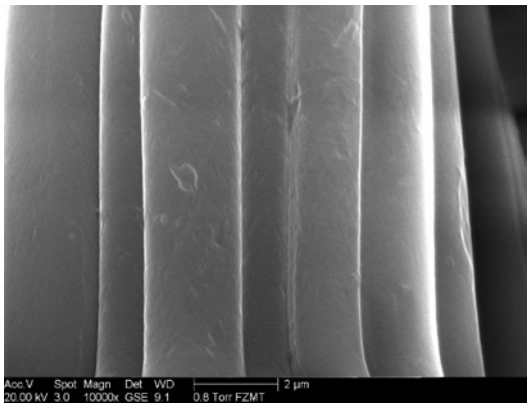
d. 3.5-1-d, magnification 100x



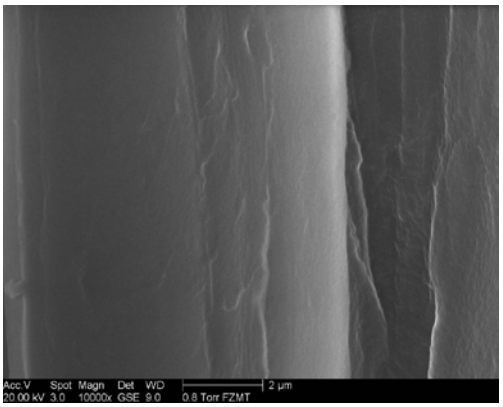
b. Untreated, magnification 1000x



e. 3.5-1-d, magnification 1000x



c. Untreated, magnification 10000x



f. 3.5-1-d, magnification 10000x

Figure 4. ESEM pictures at different magnifications (100x, 1000x, 10000x) of viscose untreated (a-c) and 3.5 mol/L (3.5-1-d) treated (d-e).

Figure 5 shows the effect of alkali concentration on the formation of reducing sugars over time during enzyme hydrolysis. The samples were processed as wet

samples without drying after 1 minute alkali treatment with cellulases. 1.0 mol/L (1.0-1-w) alkali treatment did increase hydrolysis rates to a minimal extent. At one hour hydrolysis time, 4.9 mol/L NaOH (4.9-1-w) caused the highest release in reducing sugars. After two hours hydrolysis time, cellulase hydrolysis showed a maximum at 3.5 mol/L NaOH pre-treatment (3.5-1-w). In general, the hydrolysis rate is increasing with alkali concentration of the pre-treatment step. Ammonia pre-treatment increases the degradation rate by 40% compared to the control.

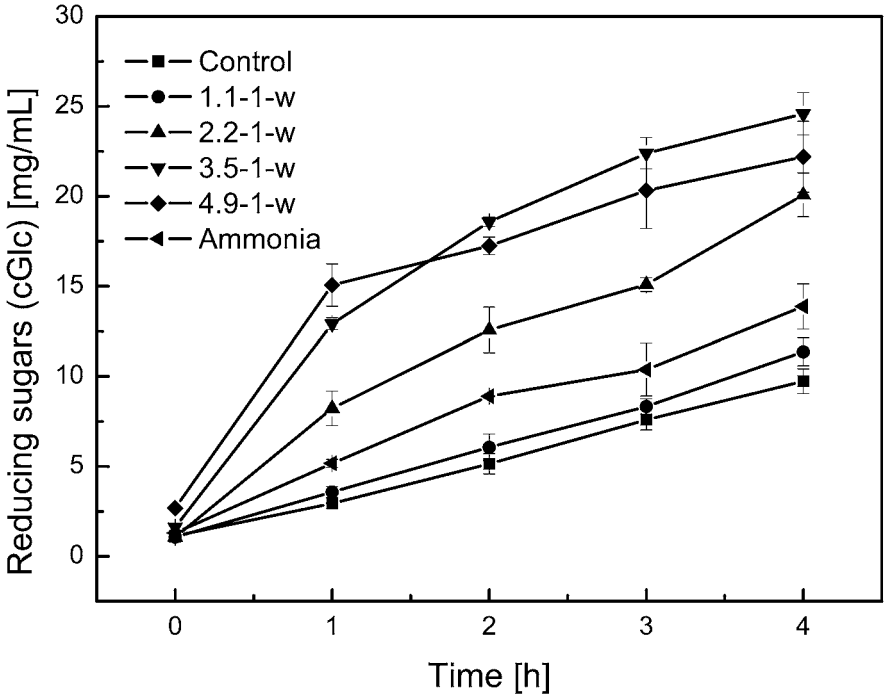


Figure 5. Release of reducing sugars at different alkali concentrations upon hydrolysis of viscose.

Also for the pre-treated sample itself an increase of reducing sugars released in liquor was found which is represented in the starting point (T = 0 h). The amount of reducing sugars found at the starting point at 4.9 mol/L (4.9-1-w) pre-treatment was about 16 times increased compared to the non-pretreated samples. This effect

can be explained by the formation of reducing sugars during aqueous alkali treatments (Klemm et al. 1998a).

Table 2 shows hydrolysis rates at the first and fourth hour of enzyme treatment. The hydrolysis rate was increasing with the alkali concentration in the first hour. This indicates an increase in accessibility of the fiber for cellulases. At four hours cellulase treatment hydrolysis rates indicated that the most accessible regions of the fiber have already been hydrolyzed by the enzyme at alkali pre-treatment concentrations above 2.2 mol/L (2.2-1-w). The increase of alkali concentration changed the hydrolysis rates mainly in the early stages of the process. At 1.0 mol/L (1.0-1-w) the hydrolysis rate remained unchanged. The maximum of total produced reducing sugars was found at 3.5 mol/L. An increase of the alkali concentration resulted in decreased overall produced reducing sugars due to slow hydrolysis rate at the end of the process where easily accessible parts of the fiber may have been removed already.

Table 2. Hydrolysis rate in the first and fourth hour of hydrolysis time in [mg/mLh] and [%] of control and total produced reducing sugars in [mg/mL] and [%] of control, depending on pre-treatment conditions.

Treatment	1st hour		4th hour		Total (4 hours)	
	[mg/mLh]	[%] of Contr.	[mg/mLh]	[%] of Contr.	[mg/mL]	[%] of Contr.
Control	3.0	-	2.2	-	8.6	-
1.0 mol/L (1.1-1-w)	3.6	120	3.0	140	10.3	120
2.2 mol/L (2.2-1-w)	8.2	280	5.0	230	19.0	220
3.5 mol/L (3.5-1-w)	12.9	440	2.2	100	23.0	270
4.9 mol/L (4.9-1-w)	12.4	410	1.9	90	19.5	230
Ammonia	3.9	130	2.1	95	12.6	150

Differences also occurred when the length of pre-treatment was varied. Figure 6 gives the weight loss after 4 hours hydrolysis time depending on alkali concentration and duration of the pre-treatment. The measured weight loss corresponds to total reducing sugars formed (calculated as released glucose) depending on the degradation rate. Samples treated two minutes with alkali showed the same trend, but a decreased weight loss, compared to samples treated for one minute. In dried samples, the effect was not as evident as for wet processed samples. 1.0 mol/L (1.1-2-w) pre-treatment resulted in almost no effect compared to control. The weight loss of ammonia treated samples was found to range between those of 1.0 and 2.2 mol/L NaOH pre-treatment. Error bars for samples treated at 2.2 and 3.5 mol/L, one minute (2.2-1-w, 3.5-1-w) are wider than those for two minute treated samples. The weight loss of the alkali treatment alone could not be determined; due to changed water uptake (measured as WRV) an increase in weight of about 1% was monitored.

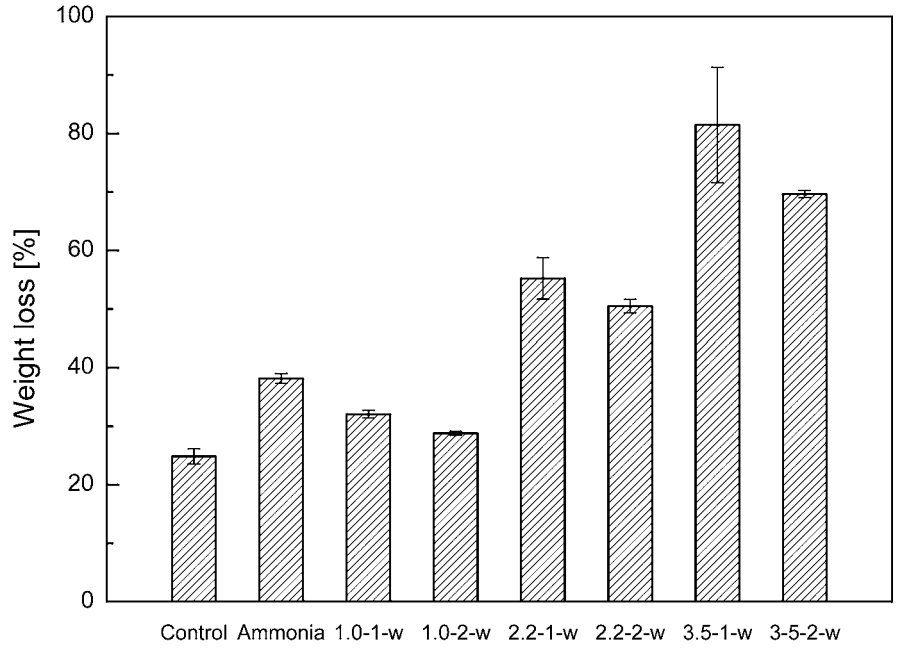


Figure 6 Effect of different alkali concentrations and alkali pre-treatment time on the hydrolysis rate of wet processed and four hours enzyme treated viscose.

The level of hydrolysis was also changed by variation of the drying conditions after alkali-treatment. Samples subjected to enzyme hydrolysis without drying (wet state) in general showed an increased degradation rate compared to dried samples, which is explained in Table 3. The differences in degradation rate between dry and wet samples increased with alkali concentration. The drying step is changing the structure of the cellulose and decreasing its accessibility and general reactivity, and therefore a decreased hydrolysis rate is obtained. The impregnation time showed a trend, but statistical analysis did not attest a significant impact for dried samples. Freeze drying did not improve degradation behavior compared to dry samples. The hydrolysis rates were found to be lower than for dried samples, but also without statistical significance.

Table 3. Weight loss [%] after 4 hours hydrolysis depending on drying conditions and duration and NaOH-concentration of pre-treatment.

		1.0 mol/L		2.2 mol/L		3.5 mol/L	
	Control	1 min	2 min	1 min	2 min	1 min	2 min
Wet	-	32.1	28.8	55.2	50.5	81.5	69.6
Line dry	24.9	26.1	25.1	43.3	43.4	52.9	50.2
Freeze dry	-	23.6	-	43.2	-	46.9	-

Figure 7 shows the amount of protein in the liquor depending on pre-treatment condition and hydrolysis time. A blank run without cellulose substrate (no substrate) showed protein denaturation to depend on process time. This is caused by many factors, such as temperature, pH and shear forces (Copeland 2000). After 4 hours treatment, all samples end up with approx. 70% remaining protein in solution. The loss of protein in solution is pronounced in the beginning and minimal at the end of the process. With increasing NaOH concentration in the pre-treatment this effect is increased. In control samples the loss of protein in solution and the reduction of activity is 30% at the end of process time. With increasing alkali concentration the loss in protein in solution and the reduction in activity by time was not found at the same extent. This indicates that only a part of the protein has cellulase activity and that only a part of the hydrolytic enzyme is involved in hydrolysis, e.g. due to surface limitations. The large scale of protein loss by high alkali concentrations supports the theory that alkalization creates easily accessible cellulose. Therefore, a greater amount of cellulases can attach to the surface which results in a reduced protein concentration in solution.

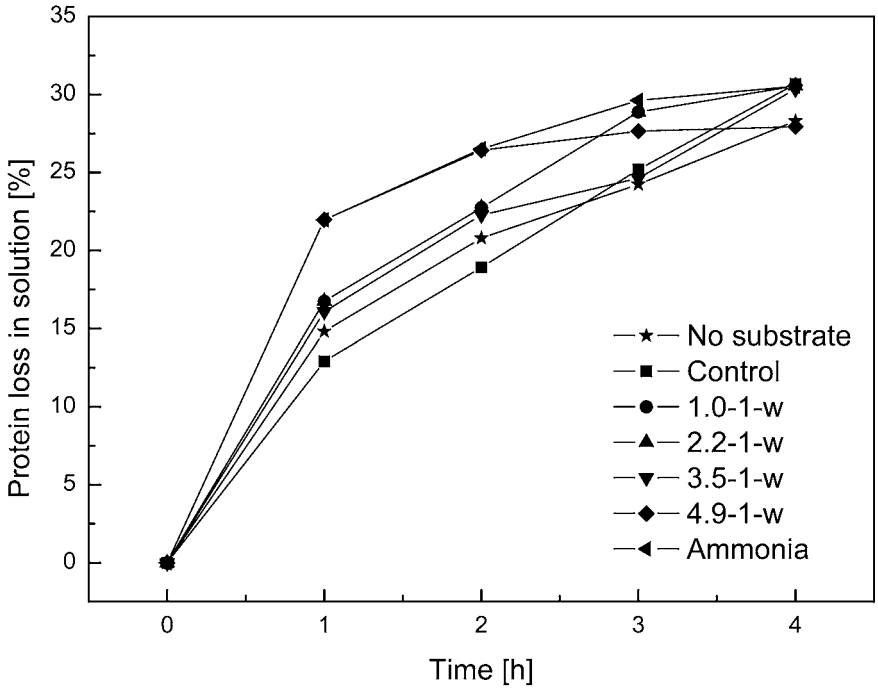
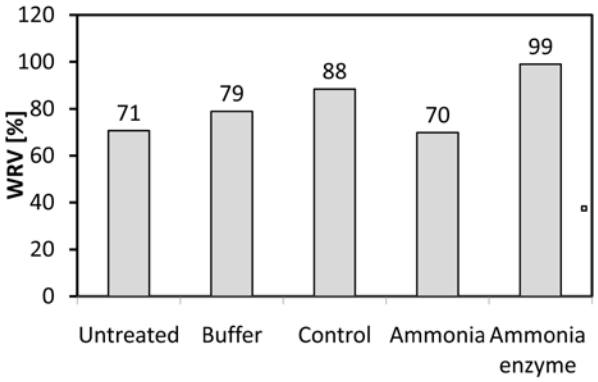


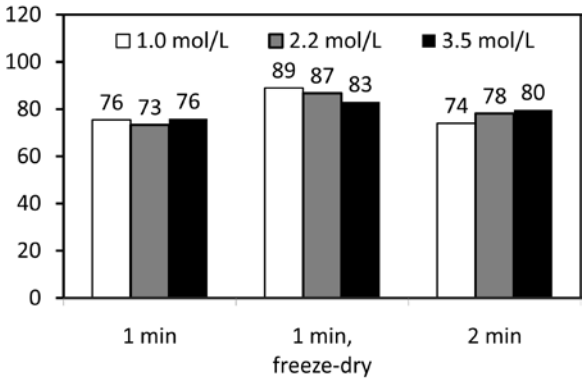
Figure 7. Protein content in treatment bath [% of initial] depending on time, substrate and pre-treatment conditions.

Figure 8 shows the water retention values (WRV) of untreated, buffer treated, control, pre-treated and enzyme treated samples. Fig 8a.: Untreated viscose fabric had a WRV of approx. 70%. Buffer treatment (enzyme process without cellulases) increased the WRV to about 80%. Enzyme treated samples showed a WRV of ca. 90%. Pre-treatment with ammonia resulted in no change to control samples, but when enzyme treated, the WRV was found to be at ca. 100%. Fig. 8b.: The samples were treated with alkali in different concentrations and line dried or, as indicated, freeze dried. No further enzyme treatment was applied. Alkali treatment resulted in WRV values at the same level as buffer treated samples. Different pre-treatment time did not have an impact on the WRV, but freeze drying showed small increases in WRV compared to control samples. The alkali concentration did not significantly

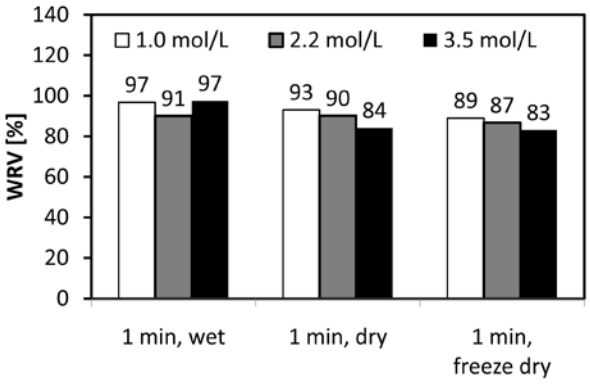
change the WRV. Fig 8c.: Samples, pre-treated for one minute with different alkali concentration, were subjected to enzyme hydrolysis. A large part of the fiber was hydrolyzed by the enzyme. The WRV in wet processed specimens was similar to ammonia treated samples, but decreased by a drying step before enzyme hydrolysis, where the WRV was found to be like in control samples. The alkali concentration had a significant impact on dried (line dried and freeze dried) samples, where the WRV decreased with increased alkali concentration. Fig 8d.: Same treatment was done as in Fig 8c, but alkali pre-treatment was performed for 2 minutes. Similar results were obtained as for 1 min pre-treated samples, but alkali concentration showed no significant trend.



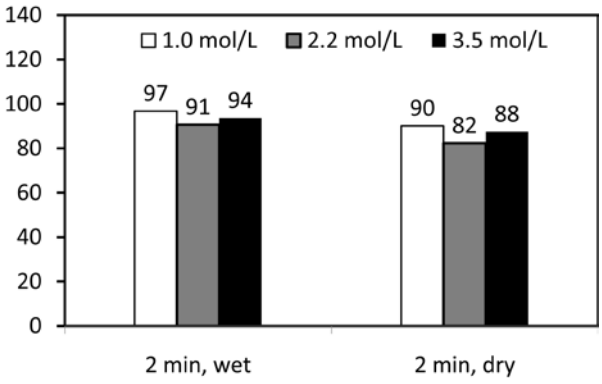
a. not pre-treated and ammonia treated



b. alkali treated



c. 1 minute alkali, and enzyme treated



d. 2 minutes alkali, and enzyme treated

Figure 8. Water retention values of samples [%]

Figure 9 shows the breaking force of 1 min alkali treated samples without enzyme treatment. Control samples showed a tensile strength of 420 kN/5 cm and 280 kN/100 yarns. In the case of alkalization, breaking force reached a maximum at 1.0 mol/L NaOH at 5 cm sample and decreases with alkali concentration. At 3.5 mol/L NaOH, 40% of the initial tensile strength was remaining. The maximum at 1.0 mol/L was obtained due to fabric shrinkage, where a greater amount of yarns per unit sample width resulted in higher strength. When calculating breaking force per 100 yarns, tensile strength was decreased by increasing alkali concentration. At 3.5 mol/L NaOH 35% of the initial tensile strength remained.

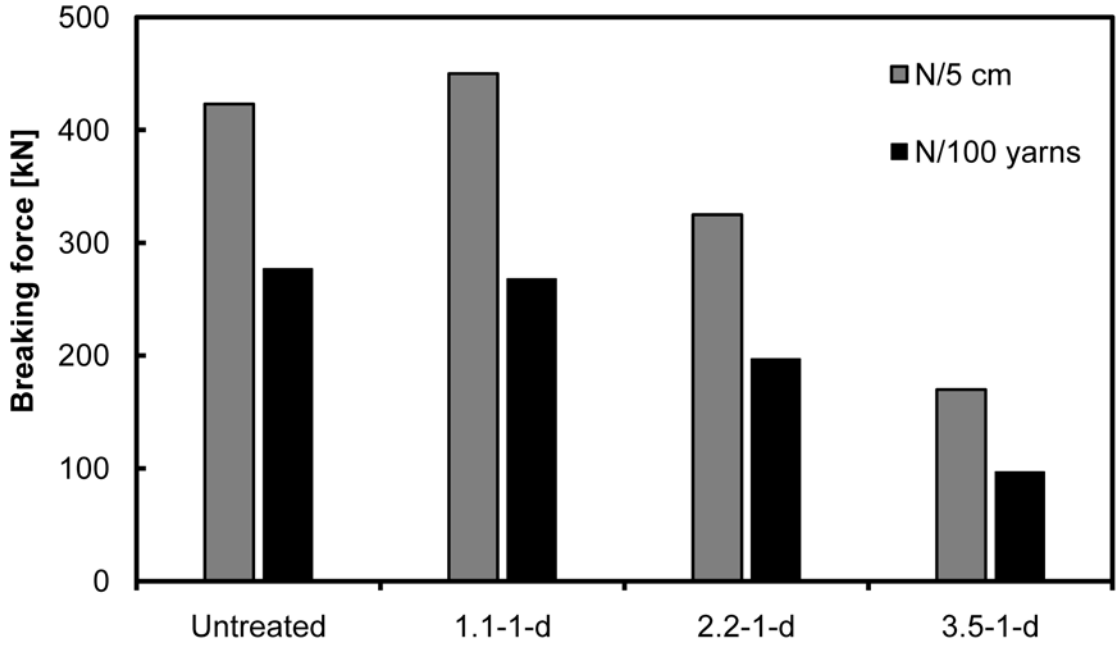


Figure 9. Breaking force of alkali treated samples depending on alkali concentration.

3.2 Local activation of the cellulose surface by printing experiments

The application and visualisation of these results was done by local surface activation on cotton textile denim fabric by alkali printing experiments. The warp of denim is ring-dyed with indigo. Enzyme treatment with cellulase leads to removal of parts of the outer – blue coloured – region of the yarns. This results in decolourisation of the fabric to a lighter shade.

Local activation of the cellulose by alkali printing enhances this effect at defined regions of the fabric. Therefore the enzyme hydrolysis causes the formation of a pattern, like in a development process of black-and-white photos.

In these experiments, the cellulase treatment caused a weight loss of the denim samples of 4% w/w for the Denim I (dark shade) and 4.5% w/w for Denim II (lighter shade). For commercial denim finishing, the treatment should be stopped at approx. 2 – 3% weight loss. This can be adjusted easily by shorter treatment time or decreased enzyme dosage. However, the ratio of colour loss between activated and reference area is not changed.

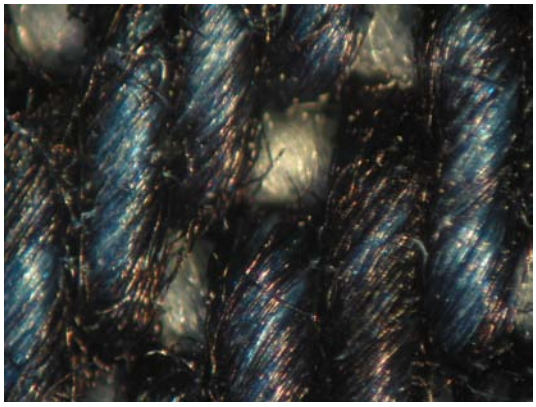
As a result of the cellulase treatment, higher amounts of indigo dyed fibres were removed from the alkali activated surface. The undyed white core of the warp yarn appears at the surface and higher contrast between surface and deeper parts of the denim fabric is observed as a result of the cellulase treatment.

The more intensive cellulase hydrolysis easily can be observed visually. Lab-values presented in Table 4 result in ΔE of about 6-6.5 for both samples.

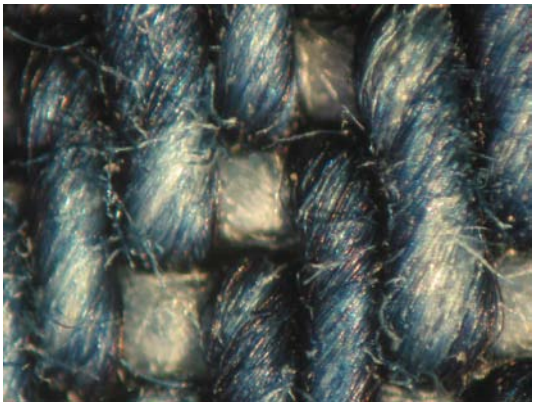
Table 4. Lab-values of Denim samples.

		Reference indigo dyed	Alkali treated, indigo dyed	ΔL / Δa / Δb	ΔE
Denim I (dark)	L	23,77	29,21	5,44	
	a	1,40	0,32	1,08	6,23
	b	-8,70	-11,54	-2,84	
Denim II (light)	L	27,76	34,08	6,32	
	a	1,34	-0,19	1,16	6,48
	b	-16,78	-15,93	0,85	

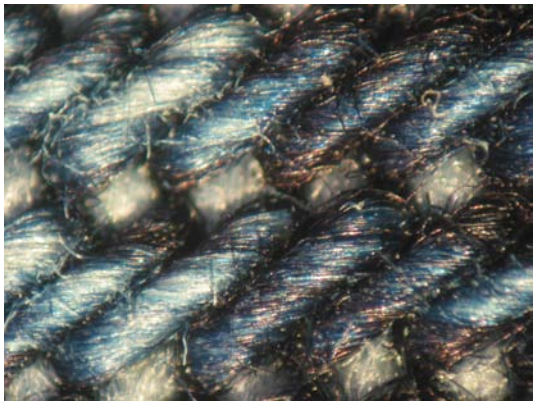
In Figure 10, microscope images of two different sites of the cellulase treated Denim I are presented. Fig. 10a shows the surface of the reference part without alkali print, in Fig. 10b the part of the same specimen is shown where the alkali paste had been printed. The difference in cellulase degradation finally resulting in a sharp line between parts of the sample is shown in Fig. 10c where alkali had been printed on (left) and not activated sections (right).



a. Reference part of sample Denim I



b. Local activation caused by alkali printing of sample Denim I



c. Border between activated and reference part of sample Denim I

Figure 10. Microscope images of reference part and alkali printed denim sample I.

4. DISCUSSION

Klemm et al. (1998b) reported pre-swelling procedures with aqueous alkali for activation of cellulose. Rath (1972) explained that usually mercerizing processes in textile industry are done at alkali concentrations of 20.6 – 26.6%. In rinsing processes, lye concentrations are lowered until all alkali is removed. Therefore an area of "maximum cellulose solubility" between 8 and 12% caustic soda is passed. In this particular range fiber damage occurs at low temperature. The experiments carried out concern the concentration range described by Rath (1972). 1 mol/L NaOH pre-treatment did not show big differences to control samples, but caustic soda treatment at 2.2 mol/L (8.8% NaOH) was located in the range of maximum fiber activation for hydrolysis. However, treatment at 3.5 mol/L created more fiber damage and hydrolysis rate which could also be determined with microscopic methods. It is assumed that the effect of fiber damage was not only created during caustic soda impregnation but also during the washing procedure. Within 4 hours, 80% of the fabric can be hydrolyzed which means almost total decomposition in a technical process. Also breaking force is decreasing with alkali concentration, where tensile strength is 3 times higher at the initial fabric than in 3.5 mol/L treated ones.

Increasing the alkali concentration did not improve degradation rate. But a greater amount of reducing sugars was released at initial stages of enzyme hydrolysis which is due to alkaline hydrolysis. However, the overall degradation rate for total hydrolysis is decreased by alkali concentrations above 3.5 mol/L.

Introducing a drying step between pre-treatment and enzyme hydrolysis was found to decrease degradability. The differences increased by increased alkali

concentration. In this set of experiments line drying and freeze drying as a very mild variant of drying were used. The fiber could not be kept in a quasi-wet-state. Overall degradation rates were decreased up to 10%. The formation of ice crystals could cause changes in the cellulose structure formed by alkali treatment. The effect could be — like increasing alkali concentration above 3.5 mol/L —, that the level of activation is reduced.

The protein content in solution was decreased during enzyme hydrolysis and ends up at approx. 70% of the initial value. Increasing the alkali concentration in the pre-treatment step caused more changes in hydrolysis rate. The change of protein content and activity during the process of the pre-treated samples indicated that only a small part of the protein had cellulase activity or was involved in hydrolysis, e.g. due to surface limitations. A marked decrease in the amount of protein in solution was found at the initial hydrolysis process by high alkali concentrations. This supports the theory that alkalization creates easily accessible cellulose regions where an increased amount of cellulases can attach to the surface and result in reduced protein concentration in the solution. A change in fiber properties caused by alkali pre-treatment does not change enzyme deactivation at the end of a 4 hours process.

The water retention value (WRV) was increased when 25% of the fiber was removed due to cellulase treatment. NaOH treatment increased the WRV by max. 25%. Variation of the alkali concentration and treatment time had no significant influence. Ammonia-treated samples showed the same WRV as untreated samples. Alkali and enzyme treatment increased the WRV by 10% – 30%. Wet processed and ammonia-treated samples showed higher WRV than dried ones. Alkali treatment and enzyme hydrolysis changed the WRV to the same degree, and have additive effects.

The increase of enzyme activity due to alkali pre-treatment is more pronounced than the increase of the WRV. Most likely, alkalization changes also pores and pore size distributions, which do not affect the WRV value. Enzyme treatment alone increases the WRV. This could support the hypothesis that cellulases create their own binding sites for further attack by loosening the crystalline region, which is initially not reflected by the WRV test.

Local activation of the cellulose surface was demonstrated with denim samples. The punctual activation was possible by application of a concentrated alkali printing paste on the fabric surface by a screen printing step. After cellulase treatment — which is required in denim finishing — differences in degradation rate were clearly visible. The change in color difference was found at a ΔE of about 6 – 6.5, independent of the initial colour depth.

5. CONCLUSIONS

In this work, the influence of caustic soda pre-treatment on degradability and changes of the fiber structure of viscose was studied. By variation of the alkali concentration and the drying conditions between pre-treatment and enzyme hydrolysis, the following can be concluded:

(I) Short (1 and 2 minutes) caustic soda pre-treatment leads to enhanced degradation rates with a maximum at 3.5 mol/L NaOH. Thereby the degradation rate can be raised by a factor of 3. Changes in the fabric structure are visible and tensile strength is reduced.

(II) A drying step after alkali-pre-treatment will reduce degradation rate markedly. Differences increase with concentration of alkali.

(III) Ammonia pre-treatment improves the degradation rate, a comparable effect will be found at concentrations of 1.0 to 2.2 mol/L NaOH.

(IV) The protein loss in liquor does not change significantly by variation of the described process conditions after 4 hours enzyme treatment. The velocity of protein loss in enzyme treatment is increased by increasing concentration of alkali in the pre-treatment step.

(V) Pre-treatment and enzyme hydrolysis change the water retention value (WRV). The alkali concentration and time do not have an influence. Wet-processed and ammonia-treated samples showed an increased water retention value, an indicator of changes in the fiber structure. Drying after pre-treatment removes the effect of increased WRV, resulting in results similar to control samples.

(VI) Local activation of cellulose surface is possible and creates clearly visible results. The process is of particular interest for the treatment of textiles with uneven distribution of dyes in the fabric, for example indigo ring dyed denim. Treatment time of finishing can be reduced, as can the costs for enzymes by decreasing the dosage. Improved physical properties are gained due to well-focused local enzyme attack whereby also new effects can be created.

ADDITIONAL INFORMATION

The practical work of this diploma thesis was performed at the Institute of Textile Chemistry and Physics at the University of Innsbruck under supervision of A.Univ. Prof. Dr. Thomas Bechtold.

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2002 – 2008	Christian Doppler Laboratory “Textile and Fibre Chemistry in Cellulosics” at the Institute of Textile Chemistry and Textile Physics, University of Innsbruck, Austria, Surface modification of cellulosic substrates with hydrolytic enzymes (e.g. cellulases)
Feb. – June 2009	Chair Wood, Pulp and Fiber Chemistry, Department of Chemistry, University of Natural Resources and Applied Life Sciences, Vienna

Practical work

Summer 1994 – 1998	Vorarlberg Milch, Feldkirch, Austria, Various jobs in production and quality control
Summer 1999, 2000	Herbert Ospelt Anstalt, Bendern, Principality of Liechtenstein Quality control in pet-food department
Summer 2000	Christian Doppler Laboratory “Pulp Reactivity” University of University of Natural Sciences and Applied Life Sciences, Vienna TEMPO-mediated oxidation of cellulose fibres
Summer 2001 – 2003	Sigma Aldrich Chemie GmbH, Buchs, Switzerland Production of biochemical products for laboratory use

Membership of professional bodies

Since 1998	Student member of Austrian Nutrition Society, Vienna
Since 2004	Student member of Association of Austrian Textile Colorists (VÖTC)
Since 2008	Alumni Association of University of Natural Sciences and Applied Life Sciences, Vienna
Since 2008	Student member of Association of Austrian Food and Biotechnologists

Publications

1 **C. SCHIMPER**, R. KECKEIS, C. IBANESCU, E. BURTSCHER, A. P. MANIAN, T. BECHTOLD, “Influence of steam and dry heat pre-treatment on fibre properties and cellulase degradation of cellulosic fibres” (DOI: 10.1080/10242420400025778), Biocatalysis and Biotransformation, Vol. **22 (5/6)**, 2004, pp. 383-389.

2 C. IBANESCU, **C. SCHIMPER**, T. BECHTOLD, "Transport properties of chemicals during drying of cellulose fabrics", Cellulose Chemistry and Technology, Vol. **38 (5/6)** , 2004 pp. 399-408.

3 **C. SCHIMPER**, T. BECHTOLD, "Mobile formaldehyde precursors in textile finishing monitored with different analytical concepts", Cellulose Chemistry and Technology, Vol. **39 (5/6)**, 2005, pp. 593-604.

4 C. IBANESCU, **C. SCHIMPER**, T. BECHTOLD, “Alkaline Treatment of Cotton in Different Reagent Mixtures with Reduced Water Content. I. Influence of alkali type and additives” (DOI 10.1002/app.22989), Journal of Applied Polymer Science, Vol. **99**, 2006, pp 2848-2855.

5 C. IBANESCU, **C. SCHIMPER**, T. BECHTOLD, “Alkaline Treatment of Cotton in Different Reagent Mixtures with Reduced Water Content. II. Influence of finishing procedure” (DOI 10.1002/app.24077), Journal of Applied Polymer Science, Vol. **101**, 2006, pp 1194-1201.

6 **C. B. SCHIMPER**, C. IBANESCU, T. BECHTOLD, “Technical Aspects in Enzymatic Hydrolysis of Cellulosics”, Lenzinger Berichte, Vol. **85**, 2006, pp 107-112.

7 **C. B. SCHIMPER**, C. IBANESCU, R. KECKEIS, T. BECHTOLD, “Advantages of a two-step enzymatic process for cotton-polyester blends.” (DOI 10.1007/s10529-007-9569-1), Biotechnology Letters, **30(3)**, 2008, pp 455-459.

8 H. M. BUI, M. LENNINGER, A. P. MANIAN, M. ABU-ROUS, **C. B. SCHIMPER**, K. C. SCHUSTER, T. BECHTOLD, “Treatment in Swelling Solutions Modifying Cellulose Fibre Reactivity – Part 2: Accessibility and Reactivity” (DOI: 10.1002/masy.200850206), Macromolecular Symposia, **262**, 2008, pp 50-64.

9 **C. B. SCHIMPER**, C. IBANESCU, T. BECHTOLD, “Effect of caustic soda on Trichoderma cellulase treatments of regenerated cellulose fibers (Part 1): Viscose”, Cellulose 2008, in press.

10 **C. B. SCHIMPER**, C. IBANESCU, T. BECHTOLD, “Localized Activation of Cellulose Fibres for Cellulase Treatment”, in preparation.

Patents

1 T. BECHTOLD, C. IBANESCU, **C. SCHIMPER**, “Verfahren zur Stabilisierung von Cellulosefasern, insbesondere einer textilen Warenbahn enthaltend Cellulosefasern“, EU-Patent 05102374.5/24.03.2005.

2 **C. B. SCHIMPER**, C. IBANESCU, T. BECHTOLD, “Cellulose Surface Activation for Cellulase Finishing”, EU-Patent 08009672.0/05.06.2007.

Scientific contributions

1 C. SCHIMPER, T. BECHTOLD, (poster) “Formaldehyd in Textilien – Formaldehyd Precursors als versteckte Emittenten in fertig ausgerüsteten Textilien – Eine kritische Auseinandersetzung“, 10. Österr. Chemietage, 17. – 19. 09. 2002, Linz, Austria.

2 C. SCHIMPER, C. IBANESCU, T. BECHTOLD, (presentation) “Ausgewählte Aspekte der Cellulasebehandlung auf Cellulosefasern”, Lyocell Workshop of Christian-Doppler Laboratory “Textile and Fibre Chemistry in Cellulosics”, 26. – 28. 03. 2003, Dornbirn, Austria. (Organizing Committee)

3 C. SCHIMPER, T. BECHTOLD, (presentation) “Formaldehyd-Precursors als versteckte Emittenten in fertig ausgerüsteten Textilien“, Lyocell Workshop of Christian-Doppler Laboratory “Textile and Fibre Chemistry in Cellulosics”, 26. – 28. 03. 2003, Dornbirn, Austria. (Organizing Committee)

4 C. SCHIMPER, R. CAMPOS, C. IBANESCU, T. BECHTOLD, (presentation) “Enzymes and Textiles – The Rate of Degradation on Different Fibres with Various Types of Cellulase-Blends”, 3rd AUTEX Conference, 25. 06. – 27. 06. 2003, Gdansk, Poland.

5 C. IBANESCU, C. SCHIMPER, T. BECHTOLD, (poster) “Migration as Basis for New Textile Effects”, 3rd AUTEX Conference, 25. 06. – 27. 06. 2003, Gdansk, Poland.

6 C. IBANESCU, C. SCHIMPER, T. BECHTOLD, (presentation) “Transport Properties of Chemicals during Drying of Cellulose Fabrics”, 13th International Symposium on Cellulose Chemistry and Technology, 03. 09. – 05. 09. 2003, Iasi, Romania.

7 C. SCHIMPER, T. BECHTOLD, (poster) “Mobile and hydrolysable formaldehyde in low-formaldehyde finishing of cellulose textile ”, 13th International Symposium on Cellulose Chemistry and Technology, 03. 09. – 05. 09. 2003, Iasi, Romania.

8 C. SCHIMPER, R. CAMPOS, C. IBANESCU, T. BECHTOLD, (presentation) “Enzymes and Textiles — The Rate of Degradation on Different Fibres with Various Types of Cellulase-Blends”, Book of Abstracts p. 30, Book of Proceedings “doc23”, 3rd Central European Conference 2003, Fibre Grade Polymers, Chemical Fibres and Special Textiles, 10. 09. – 12. 09. 2003, Portorose, Slovenia.

9 C. IBANESCU, C. SCHIMPER, T. BECHTOLD, (poster) “Migration of Chemicals During Drying Processes — Basis for Reliable Processes”, Proceedings “doc36”, 3rd Central European Conference 2003, Fibre Grade Polymers, Chemical Fibres and Special Textiles, 10. 09. – 12. 09. 2003, Portorose, Slovenia.

10 C. SCHIMPER, C. IBANESCU, T. BECHTOLD, (presentation) “Recycling Aspects in Cellulase Treatments — Savings and Process Balances”, www.vtt.fi/bel/cost847/recycling%20aspects%20handout.pdf (September 2004), COST 847 Textile Quality and Biotechnology, 2nd WG 5 meeting “Biotreatment of textile effluents”, 29. – 30. 10. 2003, Thessaloniki, Greece.

11 C. SCHIMPER, C. IBANESCU, T. BECHTOLD, (presentation) “Recycling Aspects in Cellulase Treatments — Savings and Process Balances”, www.vtt.fi/bel/cost847/mariborabstlenting.pdf (September 2004), COST 847 Textile Quality and Biotechnology, WG2 and WG 3 meeting “Bio processing of cotton and man-made fibres”, 26. – 27. 02. 2004, Maribor, Slovenia.

12 C. SCHIMPER, R. KECKEIS, C. IBANESCU, T. BECHTOLD, (presentation) “Influence of steam and dry heat 38re-treatment on fibre properties and cellulase degradation of cellulosic fibres”, Book of Abstracts “No. 35”, 3rd International Conference on Textile Biotechnology, 13. – 16. 06. 2004, Graz, Austria.

13 A. KONGDEE, C. SCHIMPER, T. BECHTOLD, (poster) “Protein fixation on cellulose substrate”, Book of Abstracts “No. P38”, 3rd International Conference on Textile Biotechnology, 13. – 16. 06. 2004, Graz, Austria.

14 C. IBANESCU, C. SCHIMPER , T. BECHTOLD, (poster) “Mercerization of cotton in alkali mixtures”, Proceedings of 4th AUTEX Conference, ISBN: 2-9522440-0-6, “O-FIB3”, 22. – 24. 06. 2004, Roubaix, France.

15 C. IBANESCU, C. SCHIMPER, T. BECHTOLD, (presentation) “Modification of cellulose properties by the use of alkali mixtures with reduced water content”, Book of Proceedings pp 385 – 391, 2nd International textile, Clothing & Design Conference – Magic World of Textiles, 03. – 06. 10. 2004, Dubrovnik, Croatia.

16 C. SCHIMPER, R. KECKEIS, C. IBANESCU, T. BECHTOLD, (poster) “Static cellulase pre-hydrolysis for batch degradation processes”, Book of Abstracts p 31, COST Action 847 & D32, 11. – 12. 11. 2004, Povoa de Varzim, Portugal.

17 C. SCHIMPER, C. IBANESCU, T. BECHTOLD. (presentation) “Total Hydrolysis of Cellulosic Fibres”, Enzyme Workshop “The use of cellulase enzymes in textile treatment and fibre characterization” of Christian-Doppler Laboratory “Textile and Fibre Chemistry in Cellulosics”, 19. – 20. 04. 2005, Lenzing, Austria.

18 C. SCHIMPER, C. IBANESCU, T. BECHTOLD, (poster) “Cellulose Fibre Influencing Cellulase Activity”, Book of Proceedings p 77, Japanese-European Workshop on Cellulose and Functional Polysaccharides, 11. – 14. 09. 2005, Vienna, Austria.

19 C. B. SCHIMPER, (presentation) “Enzymes in Textile Industry”, EPNOE Summer Course “Cellulosic Fibres” at Institute of Textile Chemistry and Physics of University of Innsbruck, 22. 08. – 25. 08. 2006, Dornbirn/Lenzing, Austria.

20 C. B. SCHIMPER, C. IBANESCU, T. BECHTOLD, (poster) “Cellulase activity depending on fibre type”, Summer Course CD, EPNOE Summer Course “Cellulosic Fibres” at Institute of Textile Chemistry and Physics of University of Innsbruck, 22. 08. – 25. 08. 2006, Dornbirn/Lenzing, Austria.

21 C. B. SCHIMPER, T. BECHTOLD, (poster) “Biotechnology in the Textile Industry – Cellulases”, Life Science Circle “Industrielle Biotechnologie – Eine Chance für Österreich?”, 16. 10. 2006, Vienna, Austria.

22 C. B. SCHIMPER, W. HARREITHER, T: BECHTOLD, (presentation) “Accessibility and reactivity of cellulases in glycerine containing solutions” 2nd International IUPAC Conference on Green Chemistry, 14. – 20. 09. 2008, Moscow, St. Petersburg, Russia.

Prizes and Awards

2003, CHT-Award 2003 - Silk textiles – care&core

The entry “silk and cotton” of FM Hämmerle to the CHT award 2003 was selected by an international jury for the first prize in the category “wellness textiles”.
The product is based on scientific results obtained at the CD-Laboratory. The research dealt with the fixation of proteins onto cellulose fibres. The successful scientific interpretation and experimental work-out of the topic was a direct result of the activity at Christian Doppler Laboratory “Cellulosics”, which offered easy access to the topic of silk-proteins. The award was presented in November 2003 in Tübingen, Germany.

2009, BOKU DOC Grants

With the aims to support young researchers and to improve the quality of dissertations at BOKU, the competitive and internationally peer-reviewed BOKU DOC grants were awarded for the first time in 2009. The entry “Novel cellulose phosphate based aerogels and its use as promising biomaterials” was awarded a funding of € 100.000 for a dissertation project of 36 months duration.

The main topic of the proposed PhD project is the synthesis of novel ultra-light aerogels from non-derivatized cellulose and cellulose phosphates with the focus on usage as cell scaffold and / or bone implant materials. The work is based on a strongly interdisciplinary approach, with 4 institutes at BOKU University Vienna and 5 national and international cooperation partners being involved in the project.

Editorial work

2003, 2005-2006 *Lenzinger Berichte* (AU-ISSN 0024-0907)

The editorial work for the Issues **82** (2003), **84** (2005) and **85** (2006) of the Lenzinger Berichte was performed together with colleagues at the Christian Doppler Laboratory “Cellulosics”, Dornbirn.

Member of commissions and committees

2008, December. Student member of the commission for habilitation of Dr. Regina Grillari, Institute of Applied Microbiology, University of Natural Sciences and Applied Life Sciences, Vienna. Topic: cell biology

2009, March. Student member of the commission for habilitation of Dr. Rainer Schuhmacher, Department of Agrobiotechnology, University of Natural Sciences and Applied Life Sciences, Vienna. Topic: analytical chemistry.

2009, June. Student member of the committee for the professorship in Nano-biotechnology with a special focus on supramolecular structures, University of Natural Sciences and Applied Life Sciences, Vienna.