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# The influence of alkali pretreatments in lyocell resin finishing – Fiber structure

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## Abstract

The fiber structure was investigated in lyocell fabrics treated with NaOH (120 g/l), or KOH (250 g/l), and compared with that in untreated fabrics. The alkali treatments caused some degree of degradation in fibers, but no significant changes in carboxyl contents or crystallinity. There were indications of transitions of porosity in the morphology of lyocell fibers, with accessibility at the fiber centers being distinctly lower than that at the outer edges. Both alkali treatments appeared to increase accessibility at the peripheral region in fibers, though to different extents; but there were no evident changes of accessibility at fiber centers. A model is proposed of changes in fiber structure due to the two alkali treatments.
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## 1. Introduction

In a previous communication (Manian, Abu-Rous, Schuster, & Bechtold, 2006), we described the influence of two *specific* alkali pretreatments on the resin finishing of lyocell – a regenerated cellulosic spun from a solution of wood pulp in *N*-methyl-morpholine-*N*-oxide. Woven lyocell fabric samples were awarded pretreatments with NaOH (120 g/l), KOH (250 g/l), or remained untreated. A half of the pretreated samples (including the untreated) were awarded resin finishing treatment by a pad-dry-cure process with a dimethylol dihydroxyethyleneurea (DMD-HEU) based product. In comparisons between samples, it was observed that: (a) there was a small but measurable increase in accessibility (measured by dye uptake) of sam-

ples pretreated with NaOH, as compared to the untreated or the KOH pretreated samples; (b) there were no significant differences in resin content between samples finished with or without pretreatment; (c) there was a greater concentration of resin on fiber/yarn surfaces in samples finished after alkali pretreatments, as opposed to a more uniform distribution of resin across fiber/yarn cross-sections in samples finished without pretreatment; (d) the crease recovery was highest in samples resin finished without pretreatment; and (e) while there were no significant differences in strength between samples subjected to pretreatments alone, both tensile strength and abrasion resistance were lower in samples resin finished after alkali pretreatment as compared to those resin finished without pretreatment.

There was little difference in wet pickup after padding, hence similar resin contents were observed in all samples. Some properties of treated samples could be correlated with resin distribution. Higher surface concentrations of

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resin result in lower abrasion resistance (Grant, Andrews, Weiss, & Hassenboehler, 1968; Lickfield et al, 2001) while a more uniform distribution of resin through substrates results in greater crease recovery (Bertoniere, King, & Rowland, 1981; Rowland, Bertoniere, & King, 1983), as observed respectively in samples resin finished with and without pretreatments. A uniform distribution of resin is also correlated with decreased tenacity, but was not found to hold true for the samples in this work. Hence, some questions remained to be answered. In particular: (i) why did alkali pretreatments result in a greater surface concentration of resin; and (ii) why was the tensile strength lower in samples resin finished after alkali pretreatments, despite a higher surface concentration of resin.

Investigations were undertaken of the fiber structure in untreated and alkali pretreated fabric samples in an effort to answer the questions raised above. In this communication, we report the results of our investigations.

## 2. Experimental

### 2.1. Alkali pretreatments

Samples of plain-woven lyocell fabric (137 g/m², 36 × 29 threads/cm, 50/1 Nm yarns comprised of 1.3 dtex fibers) were padded through treatment formulations containing alkali and surfactant. The padded samples were rolled around glass rods and enveloped in plastic sheets to be stored at room temperature: for 30 min in NaOH pretreatments, and for 4 h in KOH pretreatments. After the respective time periods, the samples were rinsed in running hot water for 5 min, immersed in a solution of 5% acetic acid at room temperature for 60 min, rinsed again in running cold water for 5 min, and line-dried overnight. The 'untreated' fabric samples received no treatments.

More details on the resin finishing of samples may be obtained from our previous communication (Manian et al., 2006). The measurements performed on the pretreated fabric samples are described below.

### 2.2. Carboxyl content

The carboxyl (COOH) content in samples was measured with the TAPPI Test Method T 237 (TAPPI Test Methods, 1989) on 0.250–0.510 g specimens cut finely into pieces of approximately 1 mm in size. Three replicate measurements were conducted on samples from each treatment.

### 2.3. IR crystallinity

The degree of crystallinity was determined from the ratio of absorbance intensities at 1370 cm$^{-1}$ to those at 2900 cm$^{-1}$ in specimens as measured by KBr transmission FT-IR spectroscopy with the method of Baldinger, Moosbauer, and Sixta (2000), who reported a good correlation between the absorbance ratio and wide-angle X-ray scattering (WAXS) values in determinations of cellulose II crystallinity. About 5 mg of finely cut fiber specimens were mixed with 1.5 g KBr, pressed into pellets, and measured in transmission mode with a Bruker IFS 66 spectrometer (Bruker, Germany). Four replicate measurements were conducted on samples from each treatment.

### 2.4. Degree of polymerization (DP)

The degree of polymerization in samples was determined by Gel Permeation Chromatography (GPC) as described by Schelosky, Roeder, and Baldinger (1999), using as eluant 0.9% (w/v) DMAc/LiCl filtered through a 0.02 μm filter. The test specimens were injected automatically, eluted through four serial GPC columns, and monitored with MALLS and refractive index (RI) detectors. The molecular weight distribution and related parameters were calculated on the basis of a RI increment of 0.140 ml/g for cellulose in 0.9% (w/v) DMAc/LiCl. Two replicate measurements were conducted on samples from each treatment.

### 2.5. Pore characterization with inverse size exclusion chromatography (ISEC)

The principle and methodology of ISEC is described in a previous communication from this laboratory (Kongdee, Bechtold, Burtscher, & Sheinecker, 2004). In essence, the material of interest is packed in a chromatography column to act as the stationary phase and probe molecules (substances of known molecular size that have no affinity for the material) are eluted through the column in a mobile phase. The accessible pore volumes are derived from the residence times of probes as shown in Eq. (1), and plotted as a function of probe diameter. The slope and y-intercept from regression analysis of the linear portion of the plot yield values of the pore parameters: total pore volume, pore surface area, and mean pore size in material as shown in Eqs. (2) and (3).

$$V_i = \frac{(T_e - T_o)F}{W} \tag{1}$$

$$V_i \approx V_p - \frac{d_i}{2}O_p \tag{2}$$

$$\bar{D}_p = \frac{V_p}{O_p} \times 2 \times 10^4 \tag{3}$$

where $V_i$ = accessible pore volume (ml/g); $T_e$ = retention time of probe (min); $T_o$ = retention time for a totally excluded component (min); $F$ = flow rate of mobile phase (0.1 ml/min); and $W$ = dry weight of material (g); $V_p$ = total pore volume (ml/g); $d_i$ = probe diameter (Å); $O_p$ = pore surface area (m²/g); and $\bar{D}_p$ = mean pore size (Å).

The experimental procedure described previously (Kongdee et al., 2004) was followed in characterizing the pore structure of samples by ISEC using as probes a series of polyethylene glycols and dextrans of increasing molecular weight. Four replicate measurements, two each on specimens from warp and fill yarns, were conducted on samples from each treatment.