Alkali Treatments of Lyocell in Continuous Processes. I. Effects of Temperature and Alkali Concentration on the Treatments of Plain Woven Fabrics

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ABSTRACT: Experiments were conducted that were designed to study the influence of various process parameters on the properties of lyocell fabrics treated with NaOH solutions in a continuous process. The process parameters of interest were the fabric type (plain, twill, or sateen woven), alkali concentration, tension on the fabric, temperature, and duration of the treatment. In this article, we present the first set of results from these experiments and examine the effect of NaOH concentration, temperature, and tension in the continuous alkali treatments of plain woven lyocell fabrics. Alkali treatments caused fiber swelling in the fabrics to extents governed by the alkali concentration and temperature of the treatment liquors. Fiber swelling caused fabric shrinkage, which resulted in changes to the fabric macroscopic structure and properties. Changes were observed in the flexural rigidity, water retention, crease recovery, strength, and abrasion resistance of the fabrics to extents governed predominantly by swelling-related changes to the fabric structure. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 3646–3655, 2009

Key words: NaOH; swelling; cellulose II; mechanical properties; processing; Tencel®

INTRODUCTION

Lyocell is a regenerated cellulosic fiber manufactured from a solution of wood pulp in *N*-methyl morpholine-*N*-oxide with a recovery of solvent exceeding 99%.[1] The lyocell process is different from that of other regenerated cellulosics, such as viscose rayon, in that it proceeds without the formation of intermediate compounds and there is no curing or ripening stage[2,3]; thereby, the fibers are imparted with a unique combination of properties. In comparison to other regenerated cellulosics, lyocell fibers exhibit a high wet strength, high wet modulus, high crystallinity, high crystallite and amorphous orientation, high wet swelling, and high fibrillation tendency.[2–6]

Cellulosic textiles are generally treated with alkali to improve their fabric properties, such as dyeability, dimensional stability, tensile strength, luster, and smoothness.[7] For lyocell fabrics, alkali treatments are also recommended as a preventive measure against the formation of permanent crease marks during wet processing.[8,9] However, alkali treatments of lyocell substrates exert a strong influence on their porosity, accessibility, reactivity, fiber splitting, fibrillation, and pilling propensities.[10,11] Previously reported investigations on the alkali treatments of lyocell substrates have involved batchwise treatments. To the best of our knowledge, there are no published reports on investigations of alkali treatments of lyocell substrates by continuous processes, which constitute a significant proportion of commercial operations.

We conducted pilot-scale experiments that simulated full-scale treatments to study the influence of various process parameters on the properties of lyocell fabrics treated with NaOH solutions in continuous processes. The process parameters of interest were the fabric type (plain, twill, or sateen woven), alkali concentration, tension on the fabric, temperature, and duration of the treatment. In this article, we present the first set of results from these experiments and examine the effect of NaOH concentration, temperature, and tension in the continuous alkali treatments of plain woven lyocell fabrics.
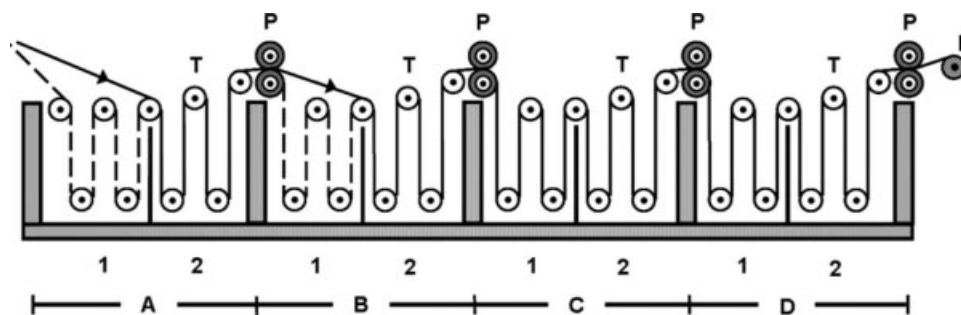
Figure 1 Schematic illustration of the washing range used in the continuous alkali treatments of the fabrics. The range consisted of four compartments (A, B, C, and D) with two subcompartments each (1 and 2), which could be heated independently; tension compensators (T); and pad or squeeze rolls (P). The solid line represents the usual route of fabric passage through the machine, whereas the dashed line represents the route used only in some instances. The fabric, after passing through the last compartment (D), was wound on a take-up roll (R).

EXPERIMENTAL

Materials

The desized and scoured 1/1 plain woven 100% lyocell (Tencel®) fabric, with a basis weight of 140 g/m² with 35 ends and 28 picks/cm and made of 30/1-Nm yarns, used in this work was kindly supplied by Lenzing AG (Lenzing, Austria). Technical-grade NaOH (ca. 50% w/w) was used to formulate the alkali solutions used in the treatments, with Lyogen MC (Clariant, Basel, Switzerland) added as wetting agent. Analytical-grade acetic acid was used to formulate the neutralization liquor.

Alkali treatments

The continuous alkali treatments were conducted on an open-width washing range, which is schematically illustrated in Figure 1. Fabric samples with dimensions of about 5×0.2 m² were subjected to successive stages of treatment, stabilization, hot washing, neutralization, and hot washing as they passed at a speed of 2 m/min through different liquors in the machine, as described in Table I, which also lists the sample residence times in the liquors (i.e., duration of treatment), the compositions and temperatures of the liquors, and the tension on the fabric. The liquor compositions and stages in the process were derived from common practices in industry.

The nip pressure in the pad or squeeze roll situated at the end of compartment A was set at 2 bar, whereas that in the other pad rolls was set at 6 bar. Free-hanging weights were suspended from the counterbalancing arms of tension compensators to achieve tensions per unit fabric width of 49 or 147 N/m in the treatment stage, 147 N/m in the stabilization stage, and 49 N/m in the stages of hot washing and neutralization/hot washing. The samples were attached at either end to suitable lengths of threader fabric (a generic polyester/cotton blend) to feed them through the washing range. The residence times were measured with a stopwatch from the time when the leading edge of the samples (or points marked on the samples) entered the liquor in one subcompartment to the time when they entered the liquor in the next subcompartment. The samples were wound on a take-up roll at the end of the range and then removed and dried by passage through a stenter heated to 130°C at a speed of 1 m/min (residence time = 60 s). Often times, the samples remained damp after they passed through the stenter and were left flat in a relaxed state to dry in ambient atmosphere.

One trial was conducted per combination of process parameters in most cases. A control experiment was conducted where a fabric sample was passed through soft water in the treatment (at 25°C) and stabilization (at 60°C) stages; this was followed by stages of hot washing, neutralization, and hot washing, as described in Table I. The tension on the fabric during the treatment stage of the control experiment was 147 N/m.

Evaluations

The samples after treatment were conditioned in a standard atmosphere of $65 \pm 4\%$ relative humidity and $20 \pm 2°C$ for 48 h before the evaluations, which were performed in the same atmosphere. The breaking force and elongation at break along the warp direction in the samples were tested with the strip method according to ISO 13934-1:1999 with a 200-mm gauge length at a 20-mm/min rate of extension. The abrasion resistance was assessed from the mass loss in the specimens after 7000 abrasion cycles, as determined according to ISO 12947-3:1999 on a Martindale abrasion tester (James H. Heal & Co. Ltd, Halifax, England), and the crease recovery angle was determined according to DIN 53890 after 30 min of recovery. The sample flexural rigidity was determined from their bending lengths measured according to BS 3356 : 1990 on a homemade apparatus. The specimens were then padded through deionized water at 2 m/min at a nip pressure of 1 bar and tested again for wet flexural rigidity. The

TABLE I
Stages in the Continuous Alkali Treatments of the Lyocell Fabrics

Stage	Compartment		Residence time (s)	Liquor[a]	Temperature	Tension[b]
Treatment	A	1	—[c]	NaOH solutions at concentrations of 90–280 g/L with 1 g/L wetting agent	Room temperature (ca. 25°C) or 40°C	49 or 147 N/m
		2	75 ± 2			
Stabilization	B	1	—[c]	NaOH solutions at concentrations of 18–56 g/L[d]	60°C	147 N/m
		2	71 ± 3			
Hot washing	C	1	65 ± 2	Water	80°C	49 N/m
		2	71 ± 3			
Neutralization	D	1	61 ± 2	1.6 g/L acetic acid	80°C	49 N/m
Hot washing		2	57 ± 1	Water	80°C	49 N/m

[a] Liquor volumes of about 20 L in each subcompartment.
[b] Tension per unit of fabric width.
[c] Fabrics were not passed through these subcompartments in this set of experiments.
[d] NaOH concentrations in the stabilization stage were targeted to be 20% of those in corresponding treatment stages.

initial weight of the specimens and their wet weight after padding were recorded to determine water pickup as a percentage of their initial weights.

To determine the water retention, about 0.5 g of each specimen was immersed for 24 h in deionized water under ambient conditions. The wet specimens were centrifuged at 4000 G for 10 min and weighed [the wet weight of the specimens (W_w; g)], dried in a laboratory oven for 4 h at 105°C, cooled to ambient temperature in a P_2O_5 desiccator, and reweighed [the weight of the specimens after drying (W_d; g)]. The water retention value (WRV; g/g) was determined with the following equation:

$$WRV = \left(\frac{W_w - W_d}{W_d} \right) \quad (1)$$

In the course of the treatments, aliquots were taken from the alkali liquors before and after the passage of the fabric samples and titrated with 0.5N HCl to determine the alkali concentrations in the liquors. Before treatment, benchmarks were drawn on the fabrics along their lengths at 20 and 15 cm apart in the warp and fill directions, respectively. The distance between the benchmarks was remeasured after treatment and drying, and the dimensional change (ΔL; %) in the fabric along the warp and fill directions was determined with the following equation:

$$\Delta L = \left(\frac{L_i - L_f}{L_i} \right) \times 100 \quad (2)$$

where L_i is the initial distance between the benchmarks (cm) and L_f is the final distance between benchmarks (cm).

The overall change in the fabric dimensions was also estimated from measurements of mass per area

on discs of diameter 38 mm obtained from the fabrics to be used as specimens in other tests.

Statistical analyses of the data were performed at a 0.05 level of significance with the software SPSS®. So the evaluation results from the untreated and control samples could be included in the plots, these samples were assigned nominal treatment liquor alkali concentrations of 62.5 and 75 g/L NaOH, respectively. There were generally no significant differences in the evaluation results between the samples treated under different tensions within one set of temperature conditions. Hence, to preserve clarity, the evaluation results were plotted as scatter plots with lines depicting the trends in values among the samples treated at 25 and at 40°C.

RESULTS

There were two primary limitations in this study: there was no mechanism to control the fabric dimensions along the fill direction during treatments, and there was no system to continuously monitor the alkali concentration in the liquors. The fabric widths were not regulated apart from efforts to prevent the samples from curling or folding at the edges.

Alkali concentrations

The alkali concentrations determined from the liquor aliquots taken before and after the passage of the samples in the liquors of different NaOH concentrations are shown in Figure 2. For the treatment liquors, the starting alkali concentrations (i.e., before the passage of the samples) are represented by the upper limits of the error bars, whereas the lower
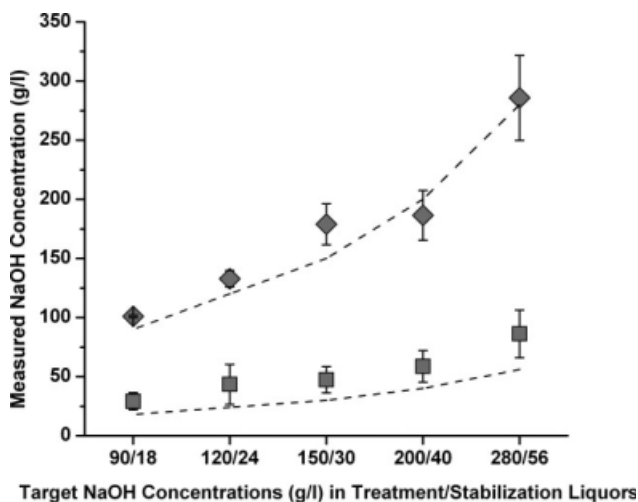
Figure 2 Concentrations of NaOH measured in the (◆) treatment and (■) stabilization liquors in the processes. The error bars denote the range between values measured before and after the passage of the samples, whereas the data points denote their average. The dashed lines denote the target values of NaOH concentration.

limits correspond to the final alkali concentrations (i.e., after the passage of the samples). For the stabilization liquors, the starting alkali concentrations are represented by the lower limits of the error bars, and the final concentrations are represented by the upper limits of the error bars. In other words, the alkali concentrations in the treatment liquors decreased whereas those in the stabilization liquors increased during the course of fabric passage in the treatments.

The differences between the target and starting alkali concentrations arose as a result of the use of technical-grade NaOH to formulate the liquors. It was not feasible to use reagents of greater purity to formulate the 20-L volumes of alkali solutions used in the experiment. Although similar amounts of fabric material were processed in all experiments, the extent of alkali depletion from treatment liquors increased with increasing alkali concentration; it increased from 1.9 to 22.3% of the initial values with increasing starting alkali concentration from 102 to 322 g/L.

The decrease in the treatment liquor alkali concentration was attributed to the sorption of alkali by the fabric, and the rise in the alkali depletion rates with increasing alkali concentration in the treatment liquor was attributed to increasing alkali sorption by cellulose with increasing alkali concentration of the solution.[12] The rise in the stabilization liquor alkali concentrations was attributed to the transport of alkali from the treatment to the stabilization liquor by the fabric and did not appear to be related to the liquor alkali concentrations. Overall, during the course of fabric passage, the stabilization liquor alkali con-

centrations increased by 55.9–66.6% of their starting values.

In view of the changing concentrations of alkali in the liquors during the treatments, the average of the starting and final concentrations, represented by the data points in Figure 2, were designated as the effective concentrations of alkali. The effective stabilization liquor alkali concentrations were 30 ± 2% of the corresponding treatment liquor alkali concentrations. All references to alkali concentrations in the following sections pertain to the effective concentrations of the treatment liquors.

The changes in the liquor alkali concentrations during the course of fabric passage caused nonuniformity in the fabric properties, which was reflected in the variability of the evaluation results and highlighted the importance of process monitoring in commercial-scale operations. However, it was possible to observe distinct influences of the process parameters on the properties of the treated fabrics, as described later.

Dimensional change

The results from the measurements of mass/area are shown in Table II, whereas those from measurements of dimensional change along the warp and fill directions are shown in Figure 3. The mass/area in the control sample was greater than that in the untreated sample, which indicated a degree of overall fabric shrinkage upon treatment with water, but it was not significantly different from those treated with 101 g/L NaOH. The fabric mass/area increased as the liquor concentrations rose to 133 g/L NaOH, did not change significantly in the range 133–186 g/L NaOH, but increased again as the concentration rose to 286 g/L NaOH. Greater overall shrinkage was observed at 25°C compared to 40°C with concentrations up to 133 g/L NaOH; at NaOH concentrations of 179 and 286 g/L, the overall shrinkage was greater at 40°C, but there was no effect of temperature on the overall fabric shrinkage with 186 g/L NaOH in the liquor.

The fill shrinkage was greater than the warp shrinkage as the fabrics were not held to length along the fill direction. The fill shrinkage increased with increasing alkali concentration up to 179 g/L NaOH and then leveled off. The greatest extent of warp shrinkage was observed with 286 g/L NaOH in the liquor. Although the fill shrinkage was generally lower at 25°C than at 40°C, peaks in the warp shrinkage were observed at both temperatures, with 133 g/L NaOH at 25°C and with 179 g/L NaOH at 40°C.

The dimensional change in the fabrics upon treatment with water (the control sample) or alkali solutions (the alkali-treated samples) was due to fiber

TABLE II
Evaluation Results

Treatment[a]	Temperature (°C)[b]	Tension (N/m)[c]	Mass/area (g/m²)	Break force (N)[d]	Elongation (%)[d]	CRA$_{(W+F)}$ (°)[e]	Mass loss (%)[f]
Untreated	—	—	143.2	732	16.9	141	9.2
Control	25	147	152.5	838	18.1	191	9.3
101/29	25	49	154.9	881	16.8	168	5.2
		147	154.1	900	17.3	175	5.8
	40	49	153.7	885	15.2	181	6.9
		147	150.5	885	15.5	188	6.1
133/43	25	49	172.6	715	16.0	109	6.9
		147	158.5	730	16.2	102	5.8
	40	49	166.5	843	20.3	158	6.8
		147	159.7	773	14.8	144	7.7
179/47	25	49	159.9	790	13.9	139	5.5
		147	164.1	748	14.7	137	6.1
	40	49	167.5	802	19.2	148	5.0
		147	164.9	812	19.6	146	5.0
186/59	25	49	164.3	741	14.7	153	5.6
		147	163.6	833	16.5	153	6.4
	40	49	163.1	839	14.8	156	5.7
		147	165.5	800	13.5	156	5.8
286/86	25	49	171.2	852	22.7	152	5.3
		147	172.0	830	22.6	146	5.2
	40	49	179.6	809	23.6	155	4.2
		147	176.3	792	23.7	162	5.9

[a] Effective alkali concentration (g/L) in the treatment/stabilization liquors.
[b] Temperature of the treatment liquor. The stabilization liquor temperature was maintained at 60°C in all experiments.
[c] Tension on the fabric (per unit of width) in the treatment stage. A fabric tension of 147 N/m in the stabilization stage was used for all experiments.
[d] Measurements along the warp direction in the fabrics.
[e] CRA$_{(W+F)}$ = crease recovery angle after 30 min of recovery, the sum of the values obtained along the warp and fill directions.
[f] Mass loss observed after 7000 abrasion cycles in the tests.

swelling, which, in turn, caused yarn swelling and led to an increase in yarn crimp and, thereby, a contraction in fabric dimensions.[8,13] The degree of fiber swelling and its effect on fabric dimensions may be influenced by factors such as alkali concentration, temperature, and mechanical restrictions imposed on fibers.

With increasing concentration of alkali in solution, the extent of cellulose swelling initially increased and then decreased, exhibiting a distinct peak.[14] The
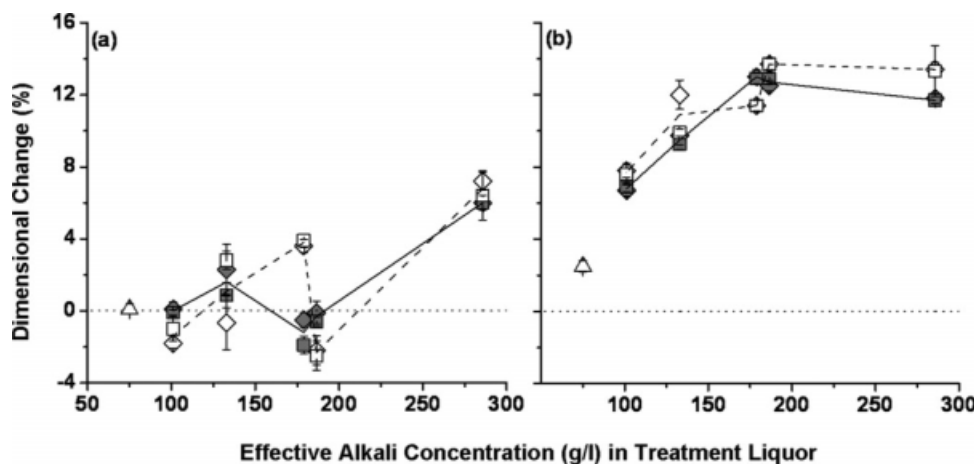
Figure 3 Dimensional change along the (a) warp and (b) fill directions in the fabrics: (△) control fabric and fabrics treated with alkali under the following treatment stage tension and temperature conditions: (■) 49 N/m and 25°C, (□) 49 N/m and 40°C, (◆) 147 N/m and 25°C, and (◇) 147 N/m and 40°C. The solid line represents the trend in values among the samples treated at 25°C, and the dashed line represents the trend among the samples treated at 40°C. The error bars denote ±1 standard deviation.

concentration of alkali at which peak swelling occurs has been shown to change with the degree of restriction on fibers; swelling peaks are observed at higher alkali concentrations for fibers within woven fabrics as compared to unrestrained fibers.[15] The rate of fiber swelling decreases with increasing alkali concentration of solutions, which is attributed to the rising viscosity and surface tension in solutions and a reduction in the rate of alkali diffusion within substrates.[15,16] With increasing temperature of alkali solutions, there is an increase in the rate but a decrease in the magnitude of fiber swelling.[15,17] The temperature also influences the alkali concentration at which peak swelling occurs in cellulosics; the concentration increases with increasing temperature.[18] In treatments with two successive alkali baths at different temperatures, the temperature of the first bath has been shown to exert a greater influence on the extent of swelling in cellulosic fibers.[19]

The greater overall shrinkage in fabrics treated at 40°C compared that in fabrics treated at 25°C in the treatment liquors with 179 g/L NaOH and higher concentrations was attributed to the effect of temperature on the fiber swelling rates. The residence time of all fabrics in the treatment liquors was maintained at 75 ± 2 s, whereas equilibrium swelling of the fibers in alkali solutions was achieved after 10–20 min.[12] Hence, it is to be expected that the shrinkage levels observed, especially in the fabrics treated at high alkali concentrations, were not a reflection of complete swelling in the fibers but that governed by rates of alkali diffusion within the substrate.

The distinct peaks in the warp shrinkage at concentrations of 133 and 179 g/L NaOH for fabrics treated at 25 and 40°C, respectively, were attributed to peak fiber swelling, as these concentrations were in the range of values commonly observed for maximum swelling in fibers.[12,15,18,20] The differences in alkali concentration at the shrinkage peaks as function of temperature may have been due to the rise in the alkali concentration of peak fiber swelling with increasing temperature.

Warp shrinkage greater than that attributed to peak fiber swelling was observed in fabrics treated with 286 g/L NaOH. The greatest levels of fill shrinkage were observed in fabrics treated with 179 g/L NaOH and higher concentrations. The overall shrinkage was also highest in fabrics treated with 286 g/L NaOH. These trends indicated higher fiber swelling in fabrics treated at alkali concentrations greater than those associated with maximum fiber swelling. It is possible that the fabric shrinkage values resulted from fiber swelling not only in the treatment stage but also in the ensuing stabilization, washing, and/or neutralization stages, with swelling extents governed by alkali concentrations within the

fabric and liquor temperatures at each stage and the alkali concentration and temperature in the first stage of treatment (i.e., in the treatment liquor).

Flexural rigidity

The results from the measurements of flexural rigidity in the fabrics are shown in Figure 4. The dry flexural rigidity was greater in the alkali-treated fabrics compared to the untreated or control fabrics. The dry flexural rigidity in the control was greater than that in the untreated fabric along the warp, but similar values were observed along the fill direction. In alkali-treated fabrics, the highest warp dry flexural rigidity was observed with 179 g/L NaOH in the liquor. Two peaks were observed in fill dry flexural rigidity: with 133 g/L NaOH in the liquor at 25°C and with 179 g/L NaOH in the liquor at 40°C. A consistent influence of temperature was not observed on the warp dry flexural rigidity of the alkali-treated fabrics. Along the fill direction, apart from the values at 179 g/L NaOH, the dry flexural rigidity was generally lower in fabrics treated at 40°C compared to those treated at 25°C.

The wet flexural rigidity was lower in the alkali-treated fabrics compared to the untreated or control fabrics. The wet flexural rigidity in the control was greater than that in the untreated fabric along the warp, but similar values were observed along the fill direction. Two peaks were observed in both the warp and fill wet flexural rigidity of alkali-treated fabrics, with 133 g/L NaOH in the liquor at 25°C and with 179 g/L NaOH in the liquor at 40°C. Apart from the two peaks, the temperature of treatment did not exert a significant influence on the wet flexural rigidity along either the warp or fill direction in the alkali-treated fabrics.

The flexural rigidity of fabrics is influenced by the ability of the yarns to move past each other at points of interlacement or crossover in the weave;[8] close contact among the yarns reduces mobility because of friction and enhances the flexural rigidity, whereas interyarn spaces enhance the mobility and reduce the flexural rigidity. In the untreated fabrics, the swelling of fibers and yarns in water reduced the interyarn spaces at crossover points, decreased the yarn mobility, and increased the wet flexural rigidity. In the alkali-treated fabrics, the increase in yarn crimp due to swelling in the alkali solutions was retained even after the yarns deswelled upon drying, and there was increased interyarn spacing at the crossover points. Upon rewetting in water, the degree of yarn swelling did not reach the levels attained during alkali treatment; hence, the interyarn spaces were maintained at the crossover points, and the wet flexural rigidity was reduced. These effects were evident in the evaluation results: the wet
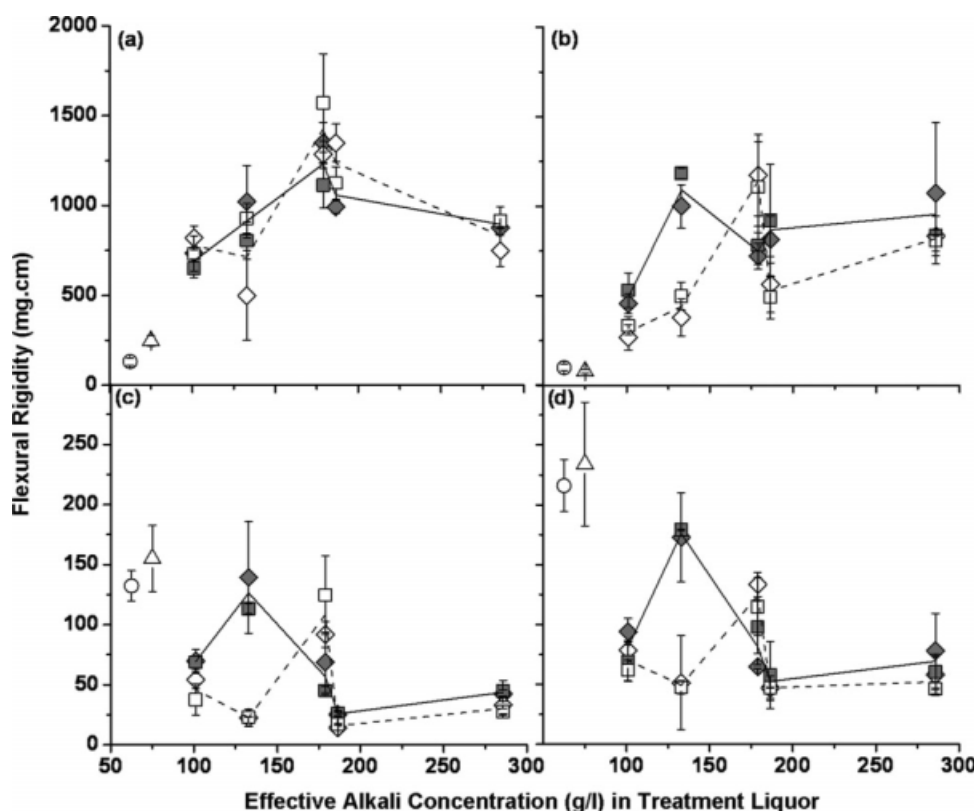
Figure 4 Flexural rigidity of the dry-state (a) warp and (b) fill and wet-state (c) warp and (d) fill-in fabrics: (○) untreated fabric, (△) control fabric, and fabrics treated with alkali under the following treatment stage tension and temperature conditions: (■) 49 N/m and 25°C, (□) 49 N/m and 40°C, (◆) 147 N/m and 25°C, and (◇) 147 N/m and 40°C. The solid line represents the trend in values among the samples treated at 25°C, and the dashed line represents the trend among the samples treated at 40°C. The error bars denote ±1 standard deviation.

flexural rigidities in the alkali-treated fabrics were lower than those in the untreated or control fabric.

The fabric flexural rigidity was also influenced by the rigidity of the yarns. High swelling in alkali solutions may have led to distortions in the shapes of the softened fibers as they became wedged against each other within yarns.[13] The fibers did not recover their original conformation even after neutralization and washing of the fabric, and the close packing of fibers within the yarns led to increased yarn and fabric flexural rigidity.[21] These effects may have been related to the maxima observed in the flexural rigidity of the fabrics treated at alkali concentrations associated with peak fiber swelling with 133 and 179 g/L NaOH at 25 and 40°C, respectively.

Fabrics awarded alkali treatments under tension, after neutralization and drying, generally exhibit high dry flexural rigidity.[13] This is attributed to a temporary setting of the fabric structure, which dissipates as fabrics are worked on in the course of further processing or handling.[22] In our study, we measured the flexural rigidities without subjecting the treated fabrics to further processing steps, and the dry flexural rigidity was measured before the wet flexural rigidity. It is possible that the high dry

flexural rigidities observed in the alkali-treated fabrics resulted from the effects of temporary fabric setting.

Water pickup and retention

The water pickup observed in the specimens when they were padded for the determination of wet flexural rigidity is shown in Figure 5. A sharp minimum in water pickup was observed in samples treated with 133 g/L NaOH, where samples treated at 25°C exhibited a lower water pickup compared to those treated at 40°C. No other significant differences in water pickup were observed between samples.

The water retention, shown in Figure 6, was greater in the alkali-treated fabrics compared to the untreated or control fabric. There were no significant differences in water retention between the untreated and control fabrics. Among the alkali-treated fabrics, there was an overall decline in the water retention with increasing alkali concentration. Two sharp minima were observed: with 133 g/L NaOH in the liquor at 25°C and with 179 g/L NaOH in the liquor at 40°C. Apart from the two minima, the temperature of the treatment liquor did not exert a significant
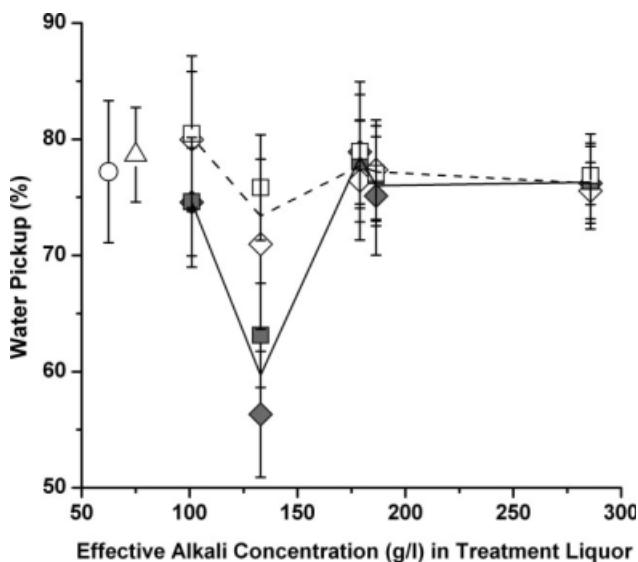
Figure 5 Water pickup in the fabrics: (○) untreated fabric, (△) control fabric, and fabrics treated with alkali under the following treatment stage tension and temperature conditions: (■) 49 N/m and 25°C, (□) 49 N/m and 40°C, (◆) 147 N/m and 25°C, and (◇) 147 N/m and 40°C. The solid line represents the trend in values among the samples treated at 25°C, and the dashed line represents the trend among the samples treated at 40°C. The error bars denote ±1 standard deviation.

influence on the water retention of the alkali-treated fabrics.

Water retention values are indicative of the water amounts held by surface tension in capillary spaces between fibers.[23] The greater water retention in the alkali-treated fabrics as compared that of to the untreated or control fabric may have been due to an increase in capillary spaces as a result of changes to the fiber conformation within the yarns in the course of swelling and deswelling during treatments. The minima in water retention observed in fabrics treated with 133 and 179 g/L NaOH at 25 and 40°C, respectively, and with 286 g/L NaOH at either temperature, may have arisen from a relative reduction in capillary spaces because of the close packing of the fibers within the yarns as a result of high fiber swelling. The comparatively low water pickup in specimens from fabrics treated with 133 g/L NaOH may have also arisen in some measure from the close packing of the fibers within the yarns in the fabrics.

Abrasion resistance

The mass losses in the abrasion resistance tests are shown in Table II. The alkali-treated samples exhibited lower mass losses compared to the untreated or control sample, which indicated greater abrasion resistance in the alkali-treated fabrics. The concentration of alkali and the temperature of the liquor did

not exert significant influences on the abrasion resistance. There were no significant differences in the abrasion resistance between the untreated and control samples.

For a given fabric, treatments that result in higher propensities for the dissipation of applied stress or for wider distribution of stress among fabric elements result in greater fabric strengths. The greater abrasion resistance of the alkali-treated fabrics as compared to that of the untreated or control fabric may have been due to a higher propensity in the treated fabrics for the dissipation of applied stress. The increased interyarn spaces at crossover points in the treated fabrics permitted higher extents of displacement among yarns, which thereby resulted in a greater dissipation of applied stress.

Break force and elongation

The values of break force and elongation at break are shown in Table II. Compared to the untreated fabric, there was a distinct increase in the fabric breaking force after control treatment. Fabrics treated with 101 g/L NaOH exhibited greater breaking force than the control fabric. The breaking force was similar among fabrics treated with 179–286 g/L NaOH but lower than that in fabrics treated with 101 g/L NaOH. A minimum in breaking force was observed in fabrics treated with 133 g/L NaOH, where samples treated at 25°C exhibited lower
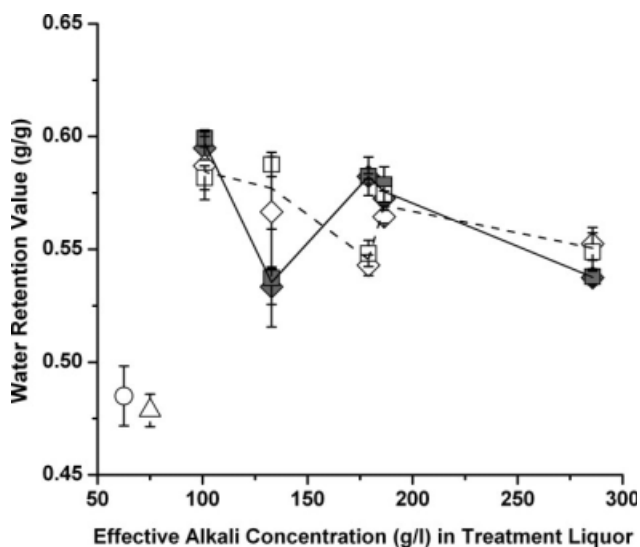
Figure 6 Water retention in the fabrics: (○) untreated fabric, (△) control fabric, and fabrics treated with alkali under the following treatment stage tension and temperature conditions: (■) 49 N/m and 25°C, (□) 49 N/m and 40°C, (◆) 147 N/m and 25°C, and (◇) 147 N/m and 40°C. The solid line represents the trend in values among the samples treated at 25°C, and the dashed line represents the trend among the samples treated at 40°C. The error bars denote ±1 standard deviation.

values compared to 40°C. No other influence of temperature was observed.

The highest elongation at break was observed in samples subjected to 286 g/L NaOH, with little difference between treatments at 25 and 40°C. In the range 101–186 g/L NaOH, the fabric elongation at break generally decreased with increasing alkali concentration; but there were differences between the samples treated at 25 and 40°C. In samples treated at 25°C, the elongation at break decreased continuously with increasing alkali concentration up to 179 g/L NaOH and increased thereafter. In samples treated at 40°C, the elongation at break decreased with increasing alkali concentration up to 186 g/L NaOH and increased thereafter, but a sharp increase in elongation was observed in fabrics subjected to 179 g/L NaOH in the liquor. The elongation at break in the untreated fabric was similar to that in fabric subjected to 101 g/L NaOH, whereas that in control fabric was marginally higher.

The alkali treatments of cellulosics may result in limited dissolution (mainly of short-length chains and hemicelluloses) and oxidative degradation of fibers,[14] and these factors may also influence the fabric strength. However, in view of the short residence time of substrates in the alkali solutions and the higher abrasion resistance in the alkali-treated fabrics, it is likely that the fabric strength was influenced predominantly by changes in the fabric structure, and any effects of fiber dissolution or degradation were relatively minor.

The rearrangements in fiber conformations within yarns in the course of swelling and deswelling during the alkali treatments influenced the propensity of stress distribution among the fabric elements. An increase in the packing density of the fibers within yarns increased the yarn rigidity, decreased the propensity of stress distribution within the fabrics, and led to reductions in the fabric strength. These factors may have been related to the minima observed in the strength of fabrics treated with 133 g/L NaOH. The greater strength in other alkali-treated fabrics appeared indicative of fiber conformations that were more favorable to stress distribution within the fabric.

An increase in yarn crimp resulted in higher fabric elongation, which was reflected in the results. The values of warp elongation, in general, paralleled those of warp shrinkage. The exception, observed in fabrics treated with 133 g/L NaOH at 25°C, may have possibly arisen from the influence of high yarn rigidity.

Crease recovery

The values of crease recovery are shown in Table II. Crease recovery in the alkali-treated fabrics decreased with increasing alkali concentration up to 179 g/L NaOH and then leveled off. A minimum in crease recovery was observed in samples treated with 133 g/L NaOH, where fabrics treated at 25°C exhibited a substantially lower crease recovery than those treated at 40°C. No other influence of temperature was observed on the crease recovery of the alkali-treated fabrics. The crease recovery in the control was similar to that in samples treated with 101 g/L NaOH, but the crease recovery in the untreated fabric was significantly lower.

Fabric crease recovery is inversely related to the rigidity and directly related to the elastic recovery in fabrics.[24] The very low crease recovery in the fabrics treated with 133 g/L NaOH at 25°C was indicative of the low elastic recovery and high rigidity in these fabrics and may have been due to high yarn rigidity. The generally low crease recovery in the alkali-treated fabrics, as compared to that in the untreated or control fabric, may have reflected the high dry flexural rigidity observed in the fabrics.

CONCLUSIONS

We examined the influence of process parameters in the alkali treatment of plain woven lyocell fabrics by a continuous process, with focus on alkali concentration, treatment temperature, and tension applied on the fabric. The alkali concentration and treatment temperature exerted significant influences on the fabric properties, but no significant influence of tension was observed in the results.

The alkali concentrations in the treatment baths decreased during the course of fabric passage through the liquors to extents that increased with increasing liquor alkali concentrations. Alkali depletion, attributed to the sorption of alkali by substrates, is of importance when one considers the process control of continuous treatments. High rates of alkali depletion will entail greater demands on process monitoring systems and will also increase the chances of variability in the properties of treated fabrics.

Peaks of fabric shrinkage were observed in samples treated with 133 g/L NaOH at 25°C and with 179 g/L NaOH at 40°C. The shrinkage peaks corresponded to peaks in the flexural rigidity and minima in water retention, crease recovery, and breaking force. The shrinkage in fabrics and corresponding changes in their properties were attributed to fiber swelling in the alkali, which influenced the fabric macroscopic structure and properties.

In the range of concentrations examined in this study, the best combination of properties was observed in fabrics treated with 101 g/L NaOH. These fabrics exhibited a low wet flexural rigidity, high water retention, high crease recovery, high abrasion

resistance, and high breaking strength compared to the untreated fabric and fabrics treated with other alkali concentrations. The extents of alkali depletion from liquor were also lowest with these treatments.

The focus in this article is the influence of alkali concentration, temperature, and applied tension on the continuous treatments of plain woven fabrics. In future communications, we will include other variables, including fabric type (plain, sateen, or twill woven) and duration of treatment. The scope of work in this article was restricted to fabric macroscopic structure and related physicomechanical properties. In a future work, we will study fabric physicochemical properties and examine the influence of alkali treatments on dyeability and other chemical treatments.

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