Effect of Processing and Reactive Dyeing on Swelling and Pore Structure of Lyocell Fibers

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Abstract

Lyocell yarns were treated with NaOH, liquid ammonia, high pressure steam and polycarboxylic acids, and dyed with five reactive dyes. The water content of the samples was also measured by the centrifugal and chromatographic techniques. The total pore volume was substantially increased by NaOH treatment, while decreased by high pressure steaming. The pore size distributions for the dyed samples were affected by the dyes used.

Introduction

Lyocell fibers show excellent tensile properties in the swollen state, although the fibers may fibrillate when they are exposed to mechanical action in the wet state. The swelling is an important factor for the fibrillation and Lenz et al.[1] reported that the high orientation of the crystallites, as well as of the less ordered inter-layers between the crystallites in the elementary fibrils, prevents the distortion of the fibrils due to the penetration of swelling agents. The swollen behavior of the processed and reactive dyed Lyocell fiber with water, however, has not been investigated in detail and is not yet well known. In this report, we first discuss the water content measured by a liquid chromatographic and centrifugal method and then the micro-pore structure of the swollen samples at different temperature are investigated with respect to variation in the processing and reactive dyeing of the Lyocell fibers.

Experimental

Commercial Lyocell (Tencel) spun yarns with 33 tex were used. Lyocell yarns were immersed in 40g/l NaOH solution at room temperature for 60 minutes. The liquid ammonia treatment was carried out for 4 seconds. The high pressure steaming was carried out at 180℃ for 5 minutes. Cross-linking treatments of Lyocell yarns were performed with a two-step method in which the samples were initially treated with 1wt% citric acid (CA) solution and then treated with 5wt% butanetetracarboxylic acid (BTCA) solution. The samples were pre-dried at 85℃ for 10 minutes, and cured at 180℃ for 2 minutes.

Five commercial reactive dyes were used: C.I. Reactive Blue 71(**B-71**), C.I. Reactive Blue 19(**B-19**), C.I. Reactive Blue 4(**B-4**), C.I. Reactive Black 5(**Bl-5**), C.I. Reactive Red 194(**R-194**). Dyeing of Lyocell yarns with the reactive dyes was carried out at a liquor ratio of 50 : 1, in the presence of 5wt% sodium chloride. Dyeing was begun at room temperature and after 20 minutes, the bath was settled to the desired dyeing temperature and 2wt% sodium carbonate was added. Dyeing continued at this temperature for 60 minutes.

Size exclusion chromatography was carried out with a system of High Performance Liquid Chromatography. The eluent was deionized water and its flow rate was 0.5 ml/min. Each bundle of Lyocell yarns was packed tightly in a stainless steel column with 6 mm inner diameter and 30 cm length. Standard polyethylene glycol (PEG) samples of different molecular weights (Mw) obtained from GL Sciences Co. Ltd. were used as the probe for measuring the pore size of swollen fibers.

Approximately 1g of Lyocell yarns was immersed in water for 1 hour at room temperature, and the yarn samples were centrifuged by using separable tubes at a speed of 3,000 RPM (1,600G) for 10 minutes at room temperature. The oven-dry weights (W) of the samples were determined after drying at 105℃. Water content W_{CF} at the present centrifugal treatment was calculated from Equation 1:

$$W_{CF} = \frac{D - W}{W} \qquad (1)$$

where D is the weight of wet fiber sample after the centrifugation.

Results

The water content W_{LC} measured by size exclusion chromatography is given by Equation 2.

$$W_{LC} = \frac{V_{swell} - V_{dry}}{W} \qquad (2)$$

where V_{swell} is the swollen volume and V_{dry} is the sample volume in dry state. Table I summarizes the results of water contents.

Table I . Water contents, W_{CF} and W_{LC} ,obtained by centrifugal and chromatographic techniques.

Sample	Water content (g/g) W_{CF}	W_{LC}
Original	0.624	0.454
a) processed fiber		
NaOH	0.634	0.472
Liquid NH_3	0.566	0.370
HP Steam	0.408	0.193
CA+BTCA	0.510	0.322
b) dyed fiber		
Blank	0.591	0.385
B-71(5%)	0.573	0.375
B-19(5%)	0.576	0.383
R-194(0.5%)	0.579	0.427
R-194(2%)	0.552	0.305
B-4(5%)	0.559	0.264
Bl-5(5%)	0.603	0.307

In order to characterize the pore size distribution, the capacity factor, k', has been generally introduced and calculated from the following equation,

$$k' = \frac{V_e - V_m}{V_m} \qquad (3)$$

where Ve is the elution volume of PEG sample and Vm is the volume of mobile phase. In our size exclusion chromatography, however, we have to consider the possible change of not only V_m but also pore structure and its volume depending on the experimental conditions. We will further normalize k' with the sample volume of swollen yarns V_{swell}, and employ k^* as follows.

$$k^* = \frac{V_e - V_m}{V_m V_{swell}} \qquad (4)$$

Fig.1 and Fig.2 show the comparisons of k^* obtained at 30℃ for processed and dyed Lyocell yarns.

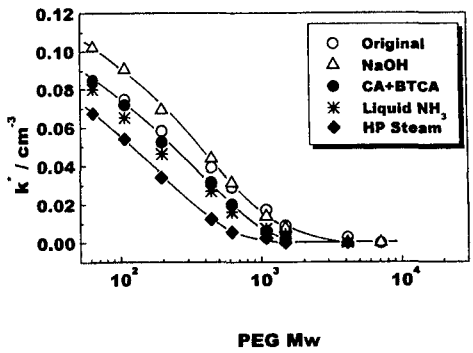

Fig 1. Comparison of k* obtained at 30℃ for processed Lyocell yarns.

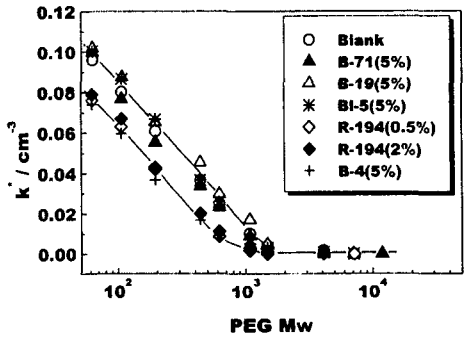

Fig 2. Comparison of k* obtained at 30℃ for dyed Lyocell yarns.

Conclusions

The degree of swelling for the processed sample was increased by NaOH treatment, while essentially decreased by polycarboxylic acids treatment and high pressure steaming. The water content and swelling of the swollen samples were also determined by the centrifugal technique. The accessible volumes of all PEG probe samples were substantially increased by NaOH treatment, while decreased by high pressure steaming simultaneously with a decrease in the permeability limit. The pore size distributions for the dyed samples at 30℃ were divided into two groups. The total pore volumes for the samples dyed with **B-71**, **B-19** and **Bl-5** were almost the same as that of the blank, while the dyeing with **R-194** and **B-4** decreased the total pore volume and the permeability limit.

References

1. J. Lenz, J. Shurz, and E. Wrentschur, Colloid Polym. Sci. 271, 460-468 (19931).