X-RAY CRYSTALLOGRAPHY OF BLEACHED AND MERCERIZED COTTONS CROSSLINKED WITH FORM W

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Extended Abstract

The authors are conducting research program to improve the absorption characteristics of cotton nonwovens [1]. They have used a continuously recording X-ray diffractometer to study changes in the crystalline structure of cotton fibers subjected to a wide range of chemical and physical treatments that include: mercerization, bleaching, crosslinking and a combination of these treatments. Mercerization involves treatment of cotton with caustic soda that swells the fibers and imparts high moisture regain and dyeability. Bleaching extracts natural impurities and makes cotton fiber white and absorbent. Crosslinking involves treatment of cotton with a chemical resin that makes the fibers more resilient. In particular, authors utilized specialized peak-profile analysis (PPA) software to study details of the changes in crystallinity induced by the crosslinking of bleached and mercerized cotton with Form W (formaldehyde).

The cotton fiber studied had a fiber length of 0.776", and a micronaire of 5.3 and was processed into a card web. The sequence of operation was: mercerization of natural fiber, bleaching of mercerized or nonmercerized natural fibers followed, finally, by crosslinking with Form W. X-ray diffraction data was obtained using a Rigaku Model D-Max B X-ray Diffractometer producing Cu K α X-rays using an accelerating voltage of 40 kV with a tube current of 60 mA.

Shadow^{©1} software was used to analyze the diffractograms of the treated cottons. An illustration of the application of peak-profile analysis is shown in Figure 1. This is an X-Ray diffractogram (solid line) of mercerized and bleached cotton showing three characteristic peaks of Cellulose II. Shadow[©] uses the whole pattern fitting technique to break the diffractogram into the sum of five component peaks shown as a dashed line. Each peak is characterized by the specific location of its center (2 θ) and the corresponding peak area (A). The area of the largest peak is called A₀. The profile will have associated a peak located at 2 θ with a normalized peak area of A/A₀. The swelling associated with mercerization causes a shift in the three principal reflection planes of the Cellulose I crystallites. Principal peak indices and locations for the Cellulose II form are as follows: [101] at 12.0°, [10<u>1</u>] at 19.8°, and [002] at 21.6°. The two additional peaks at 14.7° and 16.2° are at the same positions as the [101] and [10<u>1</u>] reflections for Cellulose I.

A summary for the results of bleaching, mercerizing, and crosslinking with Form W is given in Table 1. Results for $2-\theta$ and A/A_0 from the peak profile analysis of bleached cotton are shown in comparison to similar results from the diffractogram for cotton crosslinked with Form W. Note that both the $2-\theta$ and A/A_0 values are essentially the same for the two specimens. Thus, there is no evidence of change in the crystalline structure for bleached cotton when exposed to crosslinking. A summary of results from the peak profile analysis of bleached and mercerized cotton are also shown in comparison to results for mercerized cotton after crosslinking with Form W. It seems that the results for $2-\theta$ and A/A_0 for both cottons are quite similar. Thus, there is no evidence of change in the crystalline structure of bleached and mercerized cotton that was crosslinked with Form W.

The conclusions then are as follows:

- Crosslinking of bleached cotton with Form W does not change the crystalline (Cellulose I) nature of cotton.
- The mercerization treatment procedures used in this study yield good but slightly incomplete conversion of Cellulose I to Cellulose II.
- Crosslinking of mercerized cotton with Form W does not change the crystalline (Cellulose II) nature of the cotton.

Reference

[1] Thibodeaux, D.P. and Parikh, D.V. X-ray crystallograpy of mercerized and cemically modified cottons in nonwovens. Proceedings of Nonwovens Symposium, 1999 Beltwide Cotton Conferences, pp. 1-11. 1999.

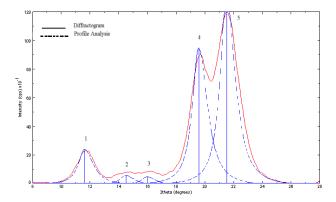


Figure 1. Results for peak profile analysis of the four treatment conditions including bleached, crosslinked with Form W, mercerized, and mercerized before crosslinking with Form W.

Table 1. X-Ray diffractogram of mercerized and bleached cotton.

Bleached		Form W		Mercerized		Mercerized/ Form W	
2-θ	A/Ao	2-θ	A/Ao	2-θ	A/Ao	2-θ	A/Ao
				11.61	18.66	11.58	36.91
14.18	22.75	14.41	25.07	14.48	5.08	14.53	8.53
16.02	20.08	16.12	19.62	15.99	4.48	16.17	11.11
				19.55	72.78	19.4	84.21
22.18	100	22.29	100	21.49	100	21.32	100

¹ Shadow[©] – A System for X-ray Powder diffraction Pattern Analysis, developed by Materials Data, Inc., Livermore, CA 94550 (1998).