

Assessment of Degree of Mercerization in Foam-Mercerized Sample

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A novel method to assess the difference in the degree of mercerization between two sides of foam-mercerized fabrics using X-ray diffraction and infrared spectroscopy is reported.

Mercerization is one of the important processes of treating cotton fabrics as it improves dyeability and strength apart from lustre. The most well-known effect of mercerization on the fine structure in cotton is the partial change of crystal lattice from cellulose I to II. During the mercerization of the cellulosic materials with about 30% alkali (w/v), the alkali breaks the bonds between various hydroxyl groups of cellulose chains in cellulose I structure and on subsequent washing and drying the structure is partially converted into cellulose II. In conventional mercerization, alkali penetrates both sides of the fabric, since the fabric is in contact with alkali on both sides. Foam mercerization^{1,2} is the latest innovation which offers the possible advantages of saving caustic soda and dyes. Foam mercerization also permits preferential treatment of one side of fabric which is not possible by conventional processes.

The degree of mercerization in a conventional mercerized sample is estimated by barium activity number (IS: 1689-1973). Foam mercerization is a highly localized effect and hence the above method is not suitable for determining the degree of mercerization in a foam-mercerized fabric. In this paper, a novel method using X-ray diffraction and infrared spectroscopy has been described to assess the difference in the degree of mercerization between the two sides of foam-mercerized fabrics.

Materials and Methods

Foam-mercerized samples were prepared using the BTRA foam technique². Foam mercerization was carried out on both bleached poplin and drill fabrics.

Sodium hydroxide solutions of 10, 12, 14, 16, 18, 20 and 25% concentrations (w/v) were prepared. Small pieces of fabric samples were immersed in slack condition in alkali solutions of various concentrations for 10 min at room temperature and washed free of alkali by the usual method and dried. These samples were used to make a calibration graph (Fig. 1).

Foam-mercerized samples were dyed with reactive dyes such as Porcion Red H8B and Orange HIR in order to determine the differential dye uptake on both sides of the fabric.

X-ray analysis—X-ray analysis of the samples was carried out on a Philips 1720 unit fitted with a vertical goniometer using nickel-filtered $\text{Cu K}\alpha$ radiation. For the diffractometer studies, the samples were finely cut, sieved through a 300 mesh screen and made into a circular pellet weighing exactly 50 mg with the aid of a special die. In the case of foam-mercerized samples, 50 mg of samples were scraped from the surface of each side of the fabric and X-ray scan was taken separately.

Infrared analysis—Infrared spectra of the treated fabrics were recorded on a Perkin-Elmer double-beam spectrophotometer (model 377) using a technique called attenuated total reflectance (ATR). The fabric was cut into the required size and kept on either side of a KRS-5 crystal capable of 10 multiple internal

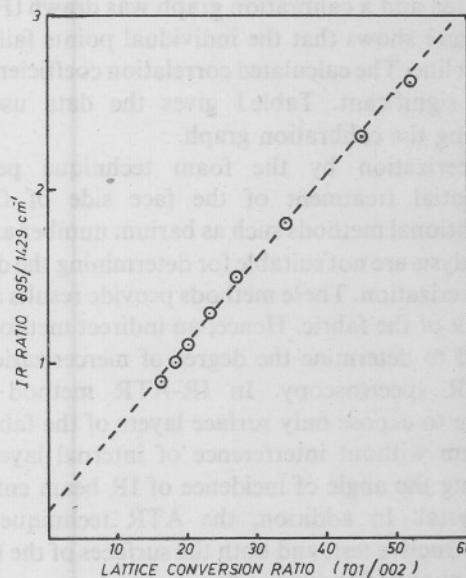


Fig. 1—Calibration plot of LCR ratio from X-ray scan against IR ratio

reflections. The crystal was mounted on the holder and the plates were screwed tightly. The sample holder was mounted on the ATR assembly and the angle of incidence of IR beam entering the crystal was adjusted to 60° , to expose only the surface layer of the fabric to the IR beam. The initial reflectance at 4000 cm^{-1} was adjusted by attenuating the reference beam to around 50%. The spectra were recorded in the region $4000\text{--}400\text{ cm}^{-1}$. In the case of the foam-mercerized sample, the spectra were taken on both sides of the fabric by reversing it.

Colour estimation—Colour difference on both sides of a dyed foam-mercerized sample was estimated using the colour matching instrument Diano Match Scan II.

Results and Discussion

Standard Calibration Curve

Treatment of cotton fabrics in alkali solution of 10% (w/v) and above at room temperature is known to effect partial conversion of cellulose I lattice to cellulose II. The amount of lattice conversion increases with increase in the concentration of alkali. The lattice conversion ratio (LCR) gives an idea of the degree of mercerization. The LCR, and hence the degree of mercerization, was estimated by the ratio of 101 and 002 peaks of cellulose II from the X-ray scan. In the case of IR scan, the relative intensities of $895\text{--}1429\text{ cm}^{-1}$ bands were calculated, an increase in the intensity of 895 cm^{-1} band and a decrease in the intensity of 1429 cm^{-1} band being indicative of mercerization.

LCR values from X-ray scans and relative intensities of $895\text{--}1429\text{ cm}^{-1}$ bands from IR scans for fabrics treated with various concentrations of alkali were calculated and a calibration graph was drawn (Fig. 1). The figure shows that the individual points fall on a straight line. The calculated correlation coefficient was highly significant. Table 1 gives the data used in obtaining the calibration graph.

Mercerization by the foam technique permits preferential treatment of the face side of fabric. Conventional methods such as barium number and X-ray analysis are not suitable for determining the degree of mercerization. These methods provide results about the bulk of the fabric. Hence, an indirect method can be used to determine the degree of mercerization by IR-ATR spectroscopy. In IR-ATR method it is possible to expose only surface layers of the fabric to IR beam without interference of internal layers by adjusting the angle of incidence of IR beam entering the crystal. In addition, the ATR technique is a nondestructive test, and both the surfaces of the fabric can be analysed individually.

IR spectra of both mercerized and unmercerized sides of foam-mercerized fabrics were taken by the IR-

Table 1—Data Used for Calibration Graph

Fabric sample slack-mercerized with	LCR from X-ray scan	IR ratio $895\text{ cm}^{-1}/$ 1429 cm^{-1}
Control (unmercerized)	0.16	0.90
10% alkali	0.18	0.98
12% alkali	0.20	1.10
14% alkali	0.23	1.30
16% alkali	0.27	1.50
18% alkali	0.34	1.80
20% alkali	0.44	2.30
25% alkali	0.52	2.60

Table 2—Data on Foam-Mercerized Samples

Foam-mercerized sample	IR ratio 895 cm ⁻¹ / 1429 cm ⁻¹	LCR from		Colour difference between two sides of fabrics
		Calibration graph	X-ray scan	
<i>Thick fabric (Drill)</i>				
Mercerized side	1.97	0.39	0.41	4.3
Reverse side	1.55	0.29	0.29	
<i>Thin fabric (Poplin)</i>				
Mercerized side	2.60	0.51	0.46	5.8
Reverse side	1.60	0.28	0.30	

ATR technique. The relative intensities of $895\text{--}1429\text{ cm}^{-1}$ bands were calculated. From the calibration graph, the corresponding LCR values were noted; this gives an indication of the amount of mercerization. To countercheck the results, the top layers of the fabric samples were scraped out, made into a pellet and X-ray scanned. LCR values were calculated directly from the X-ray scan. The experiment was repeated with the reverse side of the fabric also. LCR values obtained from the calibration graph using IR method are in good agreement with those obtained by X-ray scan directly. The results are shown in Table 2.

To investigate whether differential mercerization has taken place on both sides of the fabric, the fabrics were dyed with reactive colours and the colour difference between mercerized and reverse side was calculated. The results (Table 2) show that there is a differential dye uptake between both sides of the fabric, which indicates a difference in mercerization on both sides of the fabric.

Conclusion

Foam mercerization is a highly localized effect, and hence conventional methods such as barium activity number and X-ray diffraction cannot be used for finding out the degree of mercerization. Using IR-ATR method, it is possible to assess the degree of mercerization of both the sides of the fabric individually.

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