

## The Effect of Sodium Hydroxide on Crystallite Orientation Measurements in Cotton Fibers

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### Synopsis

The crystallite reorientation brought about by the swelling of cotton with sodium hydroxide solutions (NaOH) has been investigated. The degree of reorientation as indicated by the decreased value of x-ray angle may arise from deconvoluting and not necessarily from a change in orientation of the crystallites within the fibrillar structure.

### INTRODUCTION

Because treatment with sodium hydroxide (NaOH) has been one of the most useful chemical approaches in cotton technology, it is not surprising that the swelling of cotton with this reagent has been the subject of intensive study.<sup>1</sup> The mercerization process is known to enhance physical properties,<sup>2</sup> improve chemical reactivity,<sup>3</sup> and increase luster.<sup>4</sup> The exact mechanisms by which these changes occur are difficult to assess because of the complex structure of the cotton fiber. The x-ray angle of fiber bundles<sup>5</sup> is an accepted measure of crystallite orientation in cotton fibers. A recent publication reports the effect of convolutions upon this x-ray measurement.<sup>6</sup> The present study demonstrates the effect of sodium hydroxide swelling on convolutions and, therefore, on x-ray orientation in cotton fibers.

### EXPERIMENTAL

Approximately 0.5-g bundles of purified cotton fibers<sup>7</sup> were soaked for 1 hr at room temperature in 50 ml of various aqueous concentrations of sodium hydroxide. Solution normalities were varied in order to assess the effect of concentration on swelling. After removal from the caustic solutions, fiber bundles were rinsed in distilled water, followed by washes in 1% v/v acetic acid, 1% v/v ammonium hydroxide, and finally distilled water until neutral and then air dried.

X-Ray angles, given in Table I, were measured according to the method of Creely.<sup>5</sup> Convolution angles were measured by the optical technique of Meredith<sup>8</sup> as modified by Betrabet.<sup>9</sup>

TABLE I  
X-Ray and Convolution Angles of Sodium Hydroxide-Treated Cotton

NaOH Concentration, <i>N</i>	X-ray angle $\psi$ , °	Convolution Angle $\theta$ , °
0.0	30.8	9.2
0.5	31.5	11.4
1.0	31.0	9.9
1.5	31.7	9.7
2.0	29.2	8.5
2.5	29.5	6.8
3.0	28.2	5.1
4.0	27.4	4.3
5.0	27.3	4.3
7.0	24.9	3.3

### RESULTS AND DISCUSSION

Results are given in Table I. Linear regression analysis of the data, presented in Figure 1, yielded a correlation coefficient of +0.94.

The linear relation between x-ray angle and convolution angle implies that the increase in orientation brought about by caustic swelling may be

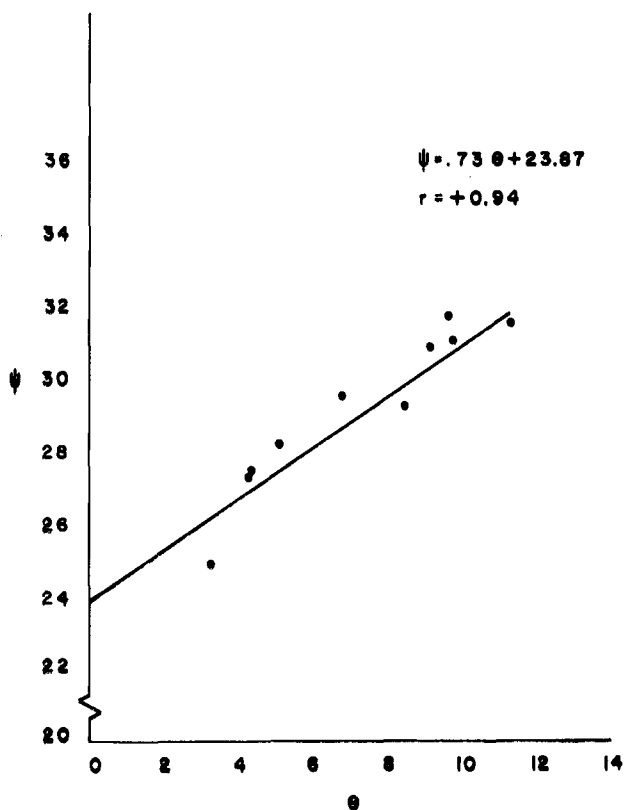


Fig. 1. Relationship of x-ray angle to convolution angle.

attributed to the reduction of convolutions. As further evidence for this position, it is interesting to note that the intercept of the regression line is very near the value of  $22^\circ$  quoted for the spiral angle of unconvoluted cotton fibers.<sup>10,11</sup> Therefore, it would seem that the increase in x-ray orientation may be a function of deconvolution and not necessarily a change in orientation of the crystallites within the fibrillar structure.<sup>12</sup>

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### References

1. J. O. Warwicker, R. Jeffries, R. L. Colbran, and R. N. Robinson, Shirley Institute Pamphlet No. 93, Shirley Institute, Manchester, 1966.
2. R. S. Orr, A. W. Burgis, F. R. Andrews, and J. N. Grant, *Text. Res. J.*, **29**, 349 (1959).
3. H. M. Spurlin, in *Cellulose and Cellulose Derivatives*, 2nd ed., E. Ott, H. M. Spurlin and M. W. Grafflin, Eds., Interscience, New York, 1954, Part 2, pp. 691 ff.
4. J. Prakash, P. G. Oka, and R. L. N. Iyengar, *Text. Res. J.*, **38**, 52 (1968).
5. J. J. Creely, L. Segal, and H. M. Ziifle, *Text. Res. J.*, **26**, 789 (1956).
6. J. J. Herbert, R. Giardina, D. Mitcham, and M. L. Rollins, *Text. Res. J.*, **40**, 126 (1970).
7. C. M. Conrad, *Ind. Eng. Chem., Anal. Ed.*, **16**, 745 (1945).
8. R. Meredith, *Brit. J. Appl. Phys.*, **4**, 369 (1963).
9. S. M. Betrabet, K. P. R. Pillai, and R. L. N. Iyengar, *Text. Res. J.*, **33**, 720 (1963).
10. J. J. Hebert, *Text. Res. J.*, **37**, 57 (1967).
11. R. Jeffries, D. M. Jones, J. G. Roberts, K. Selby, S. C. Simmens, and J. O. Warwicker, *Cell. Chem. Technol.*, **3**, 255 (1969).
12. J. O. Warwicker, *J. Appl. Polym. Sci. A-2*, **4**, 571 (1966).

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