*Effects of Mercerization on the Lateral Order Distribution of Cellulose**

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Introduction

In a previous paper[1] the authors have concluded that the fractional dissolution in sodium hydroxide solutions of various concentrations after methanolysis is the best method to obtain the lateral order distribution of cellulose. It is the purpose of the present paper to study the transition phenomena in the mercerization process using this method.

A number of investigators have studied the transition behavior in the mercerization process using the x-ray diffraction measurements[2], the moisture regain measurements[2,3] and the limit D. P. measurements[4].

In the mercerization process the phase transition of cellulose I into cellulose II accompanies a considerable decrystallization, which might have induced the discrepancy in the transition intervals observed with various measurements. So it is important to know the changes in the lateral order distribution in the mercerization process. In the present study this change was investigated with cotton linters pulp which shows a considerable change in the lateral order distribution.

1) Y. Tsuda and S. Mukoyama, This Bulletin, **29**, 748 (1956).
2) B.G. Rånby and H.F. Mark, *Svensk Papperstidning*, **10**, 374 (1955).
3) B. G. Rånby, *Acta Chem. Scand.*, **6**, 101 (1952).
4) L. Jörgensen and E. Ribi, *Nature*, **166**, 148 (1950).
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Experimental Procedure

a. Treatment with Sodium Hydroxide Solutions.— The pulp samples (1 g, powdered and bone-dried) were soaked at 20°C in 50 ml. of sodium hydroxide solutions for two hours. The pulps were recovered by suction on glass filter, washed with distilled water, N/2 acetic acid and distilled water again until they became acid-free. The samples were dried from alcohol.

b. Lateral Order Distribution.—The same method as described in the previous paper[1] was used.

c. Cellulosic Samples. — Cotton linters pulp and sulfite and sulfate wood pulps, the same samples as used in the previous study. BX yarn[5], a regenerated cellulose fibre of the highest lateral order among regenerated cellulose fibres[6].

Experimental Results and Discussion

Comparison of Lateral Order Distributions between Native and Mercerized Celluloses

In the previous paper it was shown that the lateral order distribution of cotton linters pulp is at higher order compared with wood pulps. But this difference is not observed between mercerized samples as shown in Fig. 1 and Fig. 2, at which the summative mass-order curves and lateral order distributions of both native and mercerized celluloses are shown. The distributions themselves become broad as a result of mercerization, and lower ordered portions of distributions increase. With regard to wood pulps the decrystallization accompanied by mercerization is characterized in the increase of lower ordered regions and the broadening of distributions, without appreciable changes in the lateral order of main ordered regions. However, mercerized cotton linters pulp has a similar distribution to that of wood pulp and thus the linters pulp loses its characteristic higher order after mercerization. These results mean that all of the mercerized celluloses exhibit the same lateral order distribution whatever their native origins may be. In order to compare them with regenerated cellulose fibres the lateral order distribution curve of BX yarn is also shown in Fig. 2. The lateral order of BX fibre, which is a regenerated cellulose fibre of the highest order is still lower than those of mercerized pulps, and the distribution of the former is broader than those of the latters. Therefore it will be concluded that the lateral order of regenerated cellulose is necessarily lower compared with that of native cellulose.

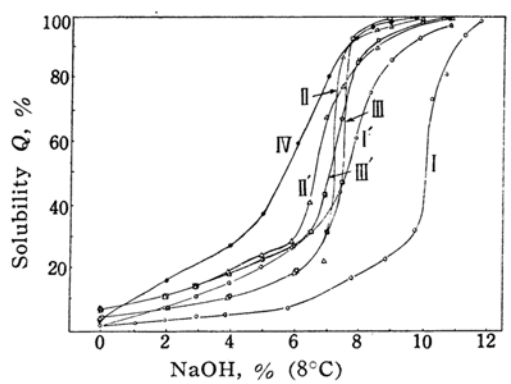

Fig. 1. Solubility curves of methanolyzed linters and sulfite and sulfate wood pulps both native and mercerized and methanolized BX yarn.
(I) native linters pulp
(I') mercerized linters pulp
(II) native sulfite wood pulp
(II') mercerized sulfite wood pulp
(III) native sulfate wood pulp
(III') mercerized sulfate wood pulp
(IV) BX yarn

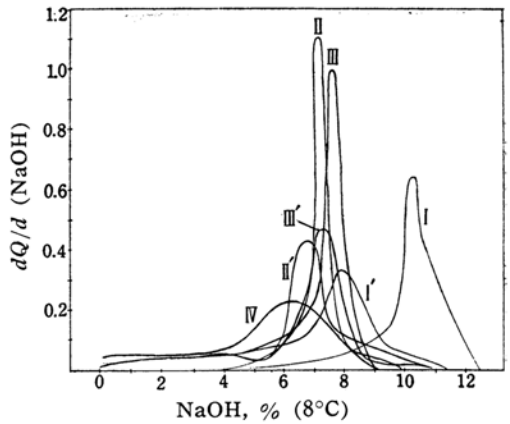

Fig. 2. Lateral order distribution curves of cotton linters and sulfite and sulfate wood pulps both native and mercerized and BX yarn.
(I) native linters pulp
(I') mercerized linters pulp
(II) native sulfite wood pulp
(II') mercerized sulfite wood pulp
(III) native sulfate wood pulp
(III') mercerized sulfate wood pulp
(IV) BX yarn

Variation of Lateral Order Distribution in the Process of Mercerization

As shown in Fig. 1 and Fig. 2, there is a remarkable variation of lateral order in the mercerization process with regard to

5) N. Drisch and L. Soep, *Text. Res. J.*, **23**, 513 (1953).
6) S. Mukoyama, *J. Soc. Textile Cellulose Ind.*,(Japan). **12**, 772 (1955).

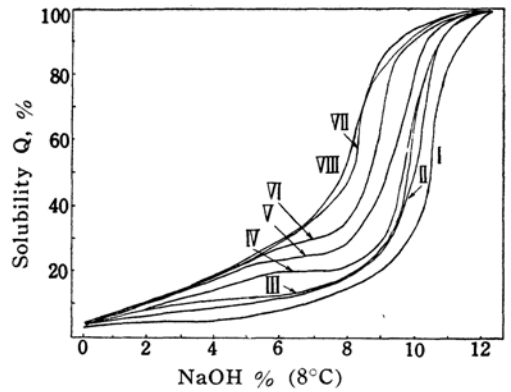

Fig. 3. Solubility curves of methanolized linters pulp pretreated with NaOH of following concentrations;
(I) original (II) 10% (III) 11% (IV) 12% (V) 13% (VI) 14% (VII) 15% (VIII) 17.5%

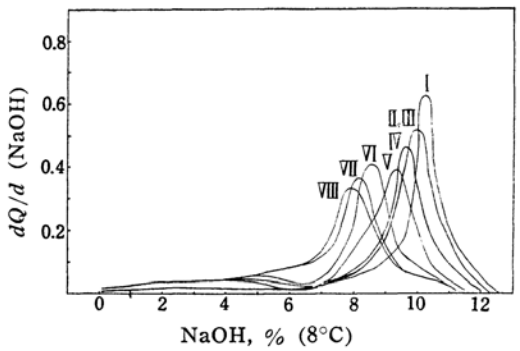

Fig. 4. Lateral order distribution curves of cotton linters pulp pretreated with NaOH of following concentrations.
(I) original (II) 10% (III) 11% (IV) 12% (V) 13% (VI) 14% (VII) 15% (VIII) 17.5%

cotton linters pulp but no appreciable variations with regard to wood pulps. Consequently as the sample for this study, cotton linters pulp was chosen. In Fig. 3 and Fig. 4 lateral order distributions of samples pretreated with sodium hydroxide solutions of various concentrations are shown. At 20°C no appreciable variation in distribution is observed up to the treatment with 10% NaOH solution. Up to the treatment with 13% NaOH solution no appreciable variations are observed in the lateral order of peak of distribution, but a successive broadening of the distribution toward the lower ordered portion can be observed with each treatment. It will be apparent in Fig. 3 and Fig. 4 that the main variation of lateral order distribution in the process of mercerization is induced by the treatment with NaOH solutions of the concentration of 13–15% at 20°C. This concentration interval of sodium hydroxide coincides with the interval which is necessary to induce phase transition. Thus the main variation of lateral order distribution is accompanied by the treatment with NaOH solutions of the concentration necessary to induce phase transition.

Here it will be possible to consider that the so-called decrystallization does not correspond to the lowering of the lateral order of the main highly ordered region but to the increase in the less highly ordered region. If these views are accepted, it will be concluded that the decrystallization is induced by the treatment with NaOH of lower concentration than that required to induce phase transition. This conclusion is consistent with the results obtained in the x-ray diffraction study as discussed in the previous paper[1].

Summary

Lateral order distributions of native and mercerized celluloses were compared and then the variation of distribution in the process of mercerization was investigated. It was concluded that mercerization generally induces broadening of distribution, and cotton linters pulp shows almost the same lateral order distribution after mercerization as those of wood pulps. The authors are grateful to Dr. K. Hoshino for permitting this publication.

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