The Structure of Never-Dried Cotton Fibers

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SYNOPSIS

The X-ray photographs of fresh never-dried cotton fibers are compared with those of dried fibers. The crystallite size of never-dried cotton fibers shows no significant correlation with maturity and orientation and is about the same as those of dried cotton fibers.

INTRODUCTION

The relationship between the structure and the mechanical properties of cotton fibers is yet to be fully established, even after many years of intensive research. The best and easiest way thought possible of linking the two is by obtaining a better understanding of the overall fiber structure, beginning with very young fibers when the structure is relatively simple and following the structure and its properties layer by layer as the cell wall is laid down till the boll opens.

Sisson[1] reported the presence of the cellulose 1 lattice even during the initial stages of the deposition of the secondary wall. Berkley and Kerr,[2] on the other hand, concluded that crystallization takes place only on drying. An investigation by Preston et al.[3] using chactomorpha cell walls showed that both never-dried and dried walls gave identically the same X-ray diffraction diagram except for the overlying water halo in never-dried walls. With cotton hairs, the water in the lumen is large relative to the wall and the water halo may completely mask the cellulose X-ray pattern.

In their study of the effect of penetration and accessibility of cotton fibers, Nelson and Mares[4] concluded that fiber crystallizes concurrently with the deposition of cellulose during the period of wall thickening. Preston[5] also found never-dried walls to be birefringent with a high degree of order in intramolecular bonding.[6] In addition, never-dried Valonia showed the same crystalline size as that in dried material. No such comparison has been carried out on cotton fibers.

The aim of this investigation is, therefore, to compare the crystallite size of never-dried and dried cotton fibers and to relate it to fiber age.

PREPARATION OF FRESH SAMPLES FOR X-RAY ANALYSIS

Fresh cotton bolls were harvested generally at 10-day intervals beginning from 20 days after flowering till the boll opened. Cotton bolls of each age were carefully removed from the plant and brought to the laboratories for X-ray examination on the same day. The husks were carefully removed with a very sharp knife to reveal the individual locks of fibers. The locks were placed in a large Petri dish. One lock at a time was placed in a separate Petri dish and single fibers separated with tweezers. Care was taken not to apply tension to each individual fiber. Combing was avoided—only a dissecting needle was used gently to parallelize the fibers. A bundle of these fibers was exposed immediately in the X-ray tube, and some were observed under the microscope. Fibers were still fresh and moist after the X-ray examination. The rest were stored in a separate receptacle (boldly labeled) to dry under room conditions. Though 20- and 25-day cottons were also examined, they were not exactly in fibrous form. They contained a large amount of water and gelatinous substances.

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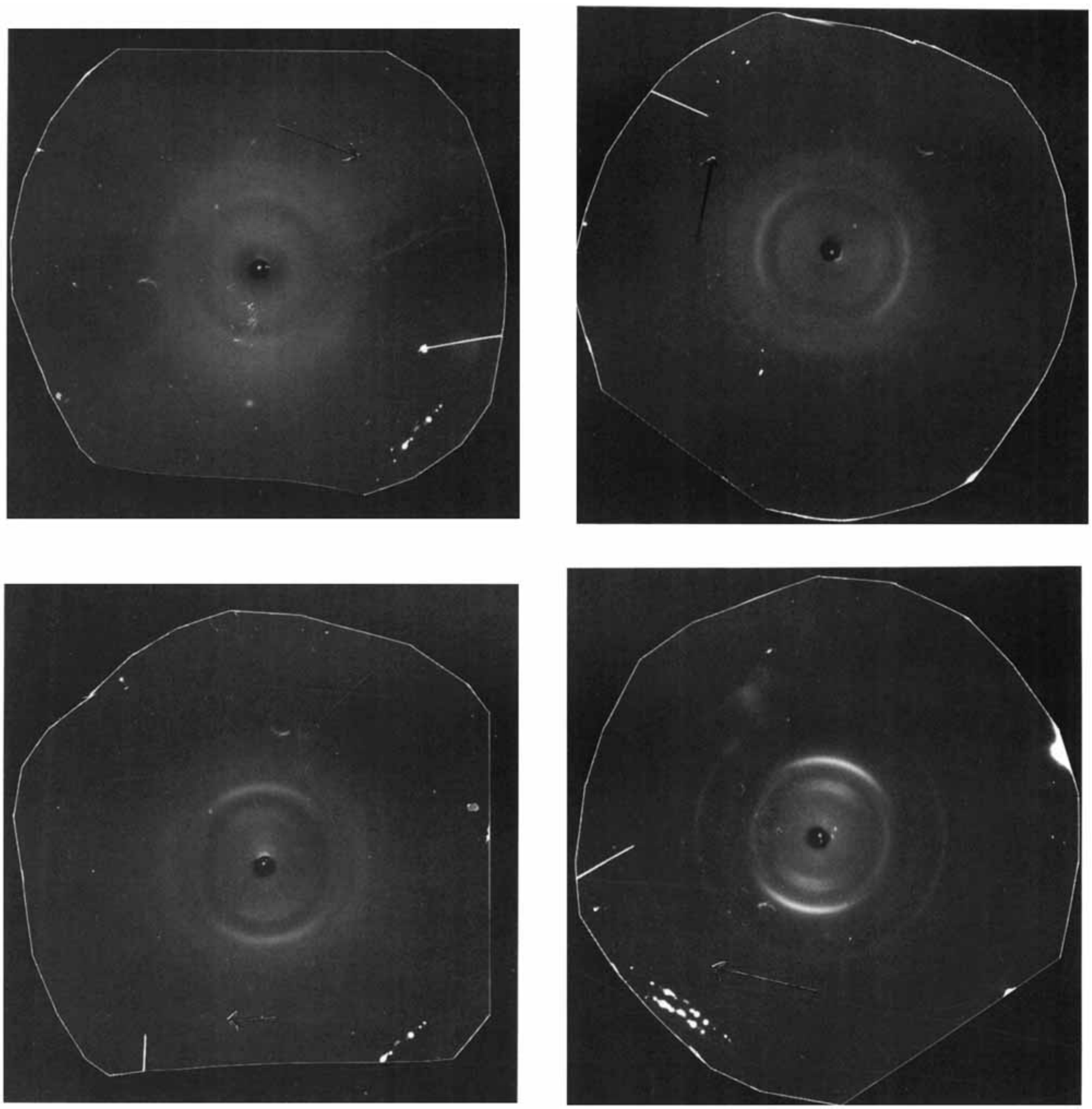
RESULTS

The X-ray photographs of the fresh fibers at different growth stages were as shown in Figures 1–4. Crystalline cellulose was not detected in 20- and 25-day-old cottons but was detected in 30-day-old cotton. The photograph of the 30-day-old cotton showed a typical cotton pattern along the (200) arc surrounded by a water halo. The 110 and $\bar{1}10$ reflections could not be clearly distinguished at this stage. Though the spread of the (200) arc showed poor orientation, it also showed that the chains were predominately in the direction of the fiber axis. These fibers were found to be birefringent, and when observed under crossed polars, no parallel extinction was observed, indicating the presence of a helical structure.

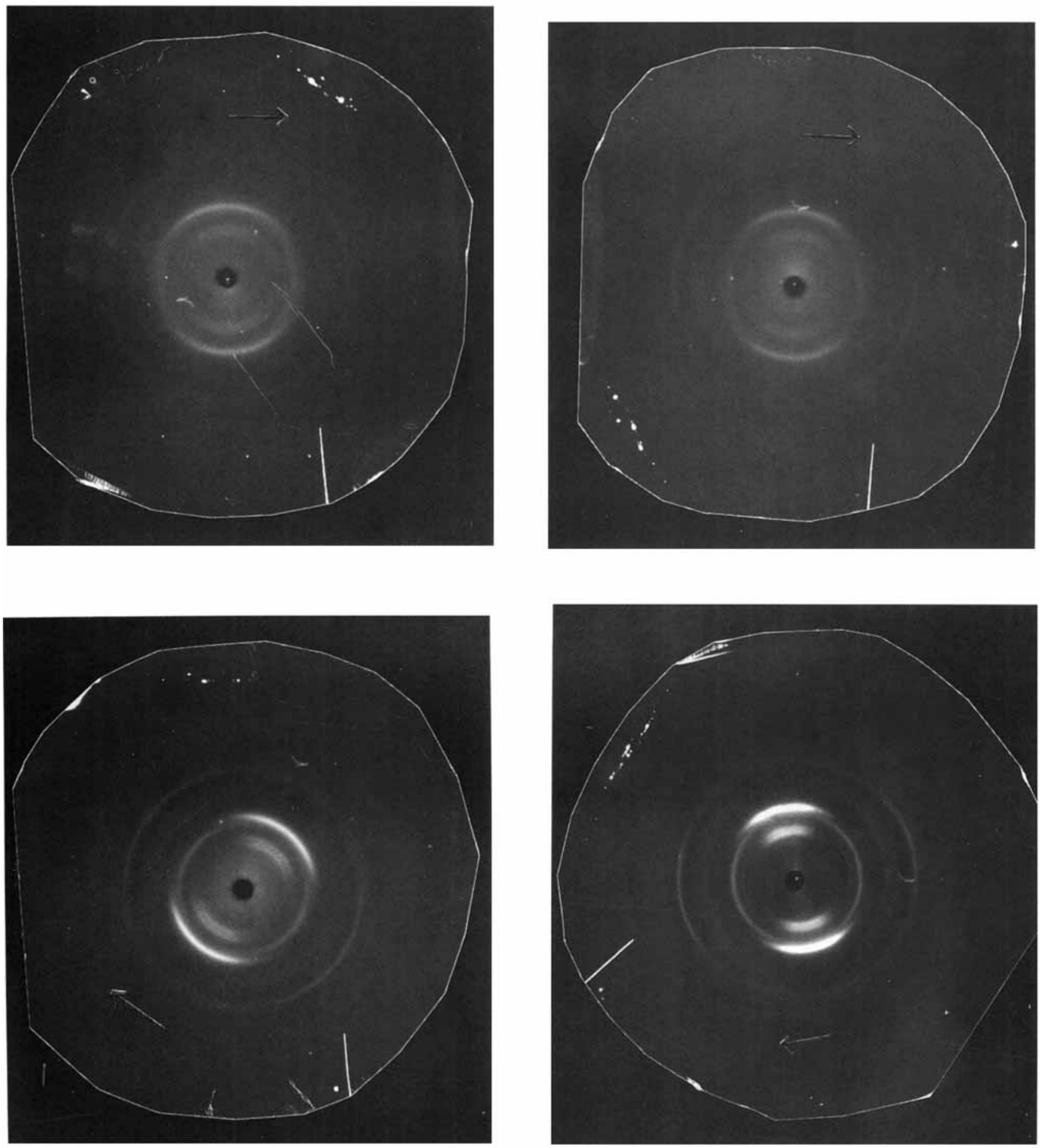
Though Berkley[7] reported that cell-wall thick-



Figures 1–4

ening started at 19 days after flowering, he also observed the usual cotton pattern at about 29 days. He observed random orientation on the 20th day. No random orientation was observed in the present work. This was probably because we did not extract these fibers with organic solvents to remove the noncellulose substances. Chemicals are known to behave differently toward young fibers, so their use was avoided in this investigation.

The overall X-ray patterns were similar for all



Figures 5–8

ages. The only noticeable differences were the gradual shortening of the arcs particularly from the (200) reflections, the corresponding increase in the intensity of the reflections, and the gradual decrease in the water content. These observations showed that the cellulose chains of the primary wall or first layer were laid down with a greater helix angle than were those in the succeeding layers. The results also showed that crystalline cellulose can be detected in fresh never-dried fibers.

The X-ray photographs of the dried fibers are similar to those of the fresh never-dried fibers with the exception of water halos (Figs. 5–8). Cuprammonium hydroxide solution was used as a running check to test for the presence of cellulose in 20- and 25-day-old dried fibers, but the fibers remained unaffected even at higher concentrations. Thirty-day-old cotton dissolves almost instantly in this solution, leaving behind crumbs of noncellulosic constituents. However, crystalline cellulose was detected in 20-day cotton grown in Nigeria, indicating the effects of climatic conditions.

50% X-Ray Angle and the Crystallite Size of Fresh and Dried Cotton Fibers at Different Stages of Maturity

The results obtained from the fresh fibers are similar to those from the dried fibers (see Table I). Table I(a) shows the results of fresh and dried Samaru cottons from the Greenhouse and Table I(b) shows the results of American Upland from the Botanic Garden.

Table I Fifty Percent X-Ray Angle and the Crystallite Size of Never-Dried and Dried Cotton Fibers at Different Stages of Maturity

Fiber Age (Days)	50% X-Ray Angle		Crystallite Size (nm)	
	Fresh	Dried	Fresh	Dried
	(a) Samaru 26J, Glasgow			
30	46	43	7.0	6.6
40	38	38	7.2	7.2
50	34	34	7.5	7.5
60	37	35	7.2	7.9
70	37	35	7.5	7.5
	(b) American Upland, Botanic Garden			
30	48	45	5.4	5.3
40	41	36	5.7	5.3
50	38	35	6.7	7.2
78	35	37	7.2	7.2

The X-ray angle from fresh unconvoluted fibers decreased steadily with age and stabilized at a mature age. This makes it more difficult to accept the view that the orientation angle in the cell wall remains constant throughout the growth period and that the X-ray angles of mature fibers are average orientation angles. The larger helical angles for immature fibers explained the flat-topped distribution.

Although the orientation in the never-dried cells must undergo certain modifications after the cell is allowed to dry, it may well be that such modification could be masked by the presence of convolutions, which is the natural state of dried cotton fibers.[8]

The crystallite size measured from both the fresh and dried cotton fibers is shown in the last column. The variations with age were found to be insignificant and the sizes of the never-dried fibers were found to be similar to those after drying. This agrees with the findings of Caulfield[9] using the Valonia plant. To explain this, we may explore the cell growth mechanism as reported by the two schools of thought. One school of thought envisages that growth in crystalline cotton occurs along what is to become the 110 crystal face: While the water recedes into the interchain space along the mass predominated (200) direction, lateral chain linkage along (200) would then result through H-bonding following gradual dehydration of the cell. The other school of thought envisages that chains may crystallize as they are deposited in the cell wall, while the water remains passively in the cell cavity. Our observations seem to follow the latter, since the (200) reflection is observed in 30-day-old cotton, which excludes the presence of intracrystalline water in never-dried fibers (Fig. 1).

CONCLUSIONS

1. The X-ray photographs of fresh, never-dried cotton fibers are similar to those of dried cotton fibers with the exception of water halos.
2. The crystallite size shows no significant correlation with maturity and orientation.
3. The crystallite size of fresh, never-dried fibers is about the same as that of the dried cotton fibers.

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