CRYSTALLINITY, CRYSTALLITE SIZE, FINE STRUCTURAL AND PHYSICAL FIBRE PROPERTIES AT TWO FIBRE MATURITY LEVELS IN TWO EGYPTIAN COTTON CULTIVARS

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Abstract

Fibre maturity is an important characteristic of cotton. It affects the yield of lint as well as the quality of cotton fibre. Two ELS Egyptian cotton cultivars, Giza 45 and Giza 77, were used in this study at 65% and 80% fibre maturity levels. The crystalline characters were studied using a wide angle X-ray diffraction technique in their native state and after waxy substance extraction. The crystallinity percent of the fibres after waxy substance extraction at high maturity cotton were 80% and 81% for Giza 45 and Giza 77, respectively; they were 67% and 69% at low maturity. Native cotton fibre gave a misleading value of 80% and 82% of crystallinity percent for high and low maturity levels of Giza 45, respectively. This result explains the importance of wax extraction during X-ray diffraction. Also, there was a high relationship between fibre maturity and degree of crystallinity after waxy substance extraction from the fibre sample. The crystallite size is almost the same for L_{002} this is about 3.5 nm for high and low maturity levels, respectively.

The cotton having higher fibre maturity had higher values of fibre strength at 1/8 or 0 inch gauge. number of convolutions per cm, and secondary wall width. The immature cotton had the higher values of waxy substances. Giza 45 was the higher in waxy substances and fibre elongation %, it had the lower ribbon width and diameter after swelling. The present study points to the importance of incorporating fibre maturity in the marketing system.

Introduction

Determining the maturity of cotton fibre as a part of the fibre testing is important for cotton yield, marketing, and textile processing. Crystallinity is known to influence the physical, mechanical and chemical behaviour of cotton. The measured crystallinity of fifteen different cultivars of cotton by the iodine - absorption method showed a very small dispersion in crystallinity, and no definite relationship between crystallinity and other physical properties (Bhujang and Najundayya, 1957). The biomodal 040 profiles given by the bundles of fully mature and fully immature fibres of Giza 45 did not show any appreciable differences. Maturity of the sample did not appear to influence the nature of the 040 profiles significantly (Chidamtareswaran *et al.*, 1975). On the other hand by using a modified X-ray diffraction procedure we found differences in crystallinity of eight cotton cultivars and that the crystallinity was correlated with fibre properties i.e, tenacity, extensibility and fineness (Hindeleh, 1980). Crystallinity ranged between 76.4 and 81.9% in *G. barbadense*, while its fineness ranged between 126 and 142 millitex (Hosemann and Bagchi, 1962).

There was a highly significant positive correlation between maturity and number of convolutions per cm (Nawar and Ghorab, 1989). Whereas, the fibre strength was correlated with the molecular weight distribution of cellulose polymers.

Crystallinity, crystallite size, fine structural and physical fibre properties, and the relationships between crystallinity and the other properties are described in the present paper.

Materials and Methods

Samples of two of ELS Egyptian cotton cultivars, Giza 45 and Giza 77, at two fibre maturity levels, were selected from the Cotton Research Institute CRI, Giza, Egypt. Waxy substances were removed by subjecting the cotton fibres to benzene extraction for six hours, washed with distilled water, boiled in 1% sodium carbonate solution for one h, rewashed and dried in an oven.

X-ray powder diffractograms for two cultivars as well as unwashed cotton fibre of the two levels of maturity of Giza 45 only, were obtained using a Philips PW 1710 diffractometer. A nickel-filtered copper X-ray tube was operated at 40 kV and 20 mA. Diffractograms of flat bundles of the fibres were recorded at a scanning speed of 1 °/min. The counter tube was fixed at the appropriate Bragg angle from either 10° to 30° or 10° to 32° of the order of 2 θ. For the air scatter trace, the cotton fibres were removed from the sample holder, the empty holder was reinserted and X-ray trace was obtained under conditions similar to those for the cotton traces.

The X-ray intensities for cotton fibres and air scatter traces were counted at 0.2 degree intervals in terms of 2 θ. Corrections for the array of intensities of cotton fibres were made for air scatter, polarisation, Lorentz factor and incoherent scatter and normalised into electron units.

The corrected and normalised array intensities were replotted against the Bragg angles and a background was simply drawn under the peaks. A polynomial equation of the form:

$$R = a+bX+cX^2+dX^3$$

was fitted; where; X is either "2 θ" or sin θ/2" (Hindeleh *et al.*, 1980), was obtained and the parameters a,b,c and d were calculated. Each peak profile was considered to have a mixed Gaussian and Cauchy function of the form:

$$F_t\, G_t + (1 - F_t)\, C_t$$

where $G_t = A_t \exp \{- \ln 2 (2 (X - P_t) / W_t)2\}$

and $C_t = A_t / \{1 + (2(X - P_t) / W_t))$

A_t, W_t and P_t are the peak parameters and F_t is the peak profile function parameter. For sensible profiles, F ranges between -0.5 and 1.0 (Hindeleh and Johnson, 1971).

In this study, an estimated value of A_t, P_t, W_t and F_t for each peak profile and the calculated background parameters (a, b, c and d) together with the corrected and normalised intensity data points and the Bragg angles "2 θ" were used as input values in a resolution program following the procedure of Powell (1964). The best agreement between observed and calculated intensities occurs when all the previous parameters are well chosen.

Crystallinity, crystallite size and lattice strain

The output data form the minimisation process and the resolution of multipeaks are obtained in the form of parameters of the resolved peaks, the area under each peak and the total area under the peaks and under the background.

The percent crystallinity is determined using the formula (Viswanthan and Shenouda, 1971).

$$\text{Degree of crystallinity \%} = \{(I_t - I_a) / I_2)\}\ 100$$

where I_t and I_a are the integral scattering interstices corresponding to the amount of total

and amorphous parts respectively.

The crystallite size normal to hkl planes "L" was calculated from the integral breadth (ib) of the peak according to Sherrers equation:

$$L = K\lambda/B \cos\theta$$

where B = ib (in radians). This was corrected for both instrumental broadening "b" and the peak profile "f" according to the mixed shape profile formula (Hindeleh *et al.*, 1980).

$$B = F (B^2 - b^2)^{1/2} + (I - F) (B - b)$$

The instrumental broadening was done using two different standard materials (Quartz and heamethylene tetramine crystals compacted at 85˚C). However the crystallite size determined using the Sherrer equation is the apparent size.

The lattice spacing d_{hkl} was determined for each resolved peak of the cotton fibres from the resolved position "P" and by using the Bragg equation.

The lattice strain "e" was calculated by using the formula:

$$e = \Delta d/d$$

where Δd is a measure of the maximum distortion in the lattice spacing "d" with respect to a standard cellulosic fibre "ramie", which is the highly crystalline form of cellulosic material "cellulose I".

True crystallite size and lattice distortion

To determine the true crystallite size, the nontransform method (Hindeleh *et al.*, 1980) was followed, in which the calculated breadth "β" is given by:

$$\beta^2 = \beta^2_S + \beta^2_D$$

where: β_S and β_D are the integral breadths due to size and distortion, which are usually defined in units of "sin θ" as:

$$\beta_S = K/L_{KKL},\ \beta_D = 4x2 \sin\theta/\lambda$$

In this analysis we used the distorted components resulting from microstrain according to Wilson (1970) and also the disorder within crystals following the paracrystalline model of Hosemann and Bagchi (1962).

Fine structural and physical fibre properties

To test fine structural fibre properties, 50 fibres of each sample were obtained. The determination of the number of convolutions per cm. and the number of reversals per cm. was made on the central 22 mm (the width of slide cover) region of fibres. The convolution angle (Fig. l) was determined from the measurement of the convolution and ribbon width adopting the formula of Sundaram (1979).

$$\tan\theta = \frac{\Pi}{2}\left(\frac{\overline{D}}{\overline{C}}\right)$$

where: D is the ribbon width, C is the pitch of the convolution and $\overline{D}$ and $\overline{C}$ are the mean values of D and C (Meredith, 1951).

Fibre strength and elongation % were determined by the Stelometer at 1/8 and 0 in. gauge. Micronaire value and mature percent were determined by IIC / FMT3. All the samples were tested under controlled atmospheric conditions at CRI, Giza, Egypt.

Results and Discussion

X-ray characteristics

X-ray diffractions traces for the two ELS Egyptian cotton cultivars Giza 45 and Giza 77 at two fibre maturity levels after waxy substances extraction (AWSE), and Giza 45 at the two levels of fibre maturity before waxy substances extraction (BWSE), and the air scatter traces were studied. For all samples, the peaks are nearly located at 2 θ = 15°, 17° and 23°, which the characteristic positions of the unresolved double 101 and $10\bar{1}$ reflections, and 002 reflection of cellulose I (Lewin and Rolex, 1975) within experimental error. Diffractograms of the low maturity (LM) and AWSE of the two cultivars revealed an additional unknown hump at about 21° unresolved from the 002 reflection.

Degree of crystallinity

All the diffractograms showed that there was considerable overlap of the diffraction peaks, specially in the BWSE fibres, so that peak resolution was the only valid method for obtaining reliable parameters for measuring crystallinity and apparent crystallite size. The two levels of BWSE and AWSE of Giza 45, were distributed into three peaks 101, $10\bar{1}$ and 002, and also a background scatter. The two levels of maturity of Giza 77 were distributed also into three peaks and a background scatter. There were some discrepancy between observed and calculated intensities. The best agreement, however, was accomplished by adding a fourth peak parameter in the peak resolution program.

Details and the significant parameters for all cotton samples BWSE and AWSE after resolving into three peaks and background are given in Table 1. LM cotton profiles resolved onto 4 peaks and background (Table 2). The fourth peak (noted as para) occurs at 21.3° (2 θ) for LM sample of Giza 45 and at 20.5° (2 θ) for LM sample of Giza 77 with different area percent. This fourth peak may represent the liquid-like scattering of cellulosic materials not in the crystalline registry. This fourth peak occurred only in LM cotton fibres, where the molecules did not have the chance to arrange themselves properly and is considered as an imperfection in the crystal lattice.

Hosemann and Bagchi (1962) showed that the imperfection in crystal lattice gives a background scatter in addition to the Bragg reflection of the crystal. Hermans and Weidinger (1949) attributed the background scatter to the amorphous regions. On the other hand. Hindeleh and Johnson (1978) explained the background scatter as a summation of Hosemann's and Herman's background scatter. Therefore, the fourth peak can be taken to represent the scattering by the amorphous material and its area is added to the background scattered area as mentioned in some earlier work (Khalifa *et al*., 1991).

The addition of such peak to the background area caused some drop in the degree of crystallinity percent so that we obtained sensible results in comparison with HM cotton fibres and in good agreement with other published results. Table 3 shows that the degrees of crystallinity of HM cotton fibres were 80% and 81% for Giza 45 and Giza 77; respectively, they are 67% and 69% for LM in the same order. Meanwhile the degree of crystallinity of BWSE cotton fibre gave a misleading value of 80% and 82% for the HM and LM of Giza 45

cultivar. This could be explained by the waxy substances that coat the fibres, which appear much more in the powder of immature fibres than those of the powder of mature fibres, which has the same weight, such that the LM fibres have less hair weight and more specific area of waxy substances. Giza 45 recorded high values of waxy substances, in contrast, low values of this property were recorded by Giza 77. The results showed that by extracting fibres with benzene, the content of waxy substances in white cotton fibre averaged 0.5 - 0.6% or moisture free weight. In addition waxy substances decreased as the degree of cellulose increased (Sandor *et al*., 1973). The degree of crystallinity percent of LM fibres were quite comparable with the published values for some Egyptian cultivars which vary between 53% and 69% (Hindeleh, 1980) .

Regarding L_{002}/d_{002}, did not change either with the level of maturity or by the effect of waxy substance extraction, while both Llol/dlol and Llol-/dlol- were slightly affected. From Tables 4 and 5, it could be seen that neither the apparent crystallite size nor the number of diffraction planes constructing the crystallites, may be used to differentiate between levels of maturity.

To see whether the integral breadth calculated by considering both microstrain and paracrystalline theories of disorder make a difference, we determined the true crystallite sizes of various cotton samples. Table 6 shows the recalculated values of L_{101}, L_{101}- and L_{002} for low and high maturity cotton samples considering both theories. from Table 6, it could be noted that both strain and paracrystalline distortion factor-for the L_{002} reflection are lower than those with the 101 and 101^{-}. Although L_{101} and L_{002} for LM are lower than those for HM, whereas L_{101}- are higher for LM than those of HM fibres.

Maturity and crystallinity

Data pertaining to the fibre maturity of the two cultivars at the two levels of maturity are shown in Table 6. The "r" value of the relationship between maturity and with crystallinity Table 7 was 0. 997. The presence of excessive immature fibres in cotton indicate some defect in the- plant growth, either varietal or environmental. It follows that cotton fibre maturity can be thought to be a composite of several structural features including crystallinity. This result illustrates the importance of fibre maturity, it is an index of the extent of development of the fibre. The degree of secondary thickening will cause more yield of lint. Also, immature cotton produces yarns with high number of imperfections and thus substandard textile products. The goal of producing strong, fine and mature cotton is becoming a reality. As the demand for those cottons increase, new and improved cultivars are being developed to supply this demand. Experience has shown that breeders can develop cotton that will satisfy the needs of the textile manufacturing industry (Gannaway, pers. comm.). The goal is to increase the number of fibres in the cross - section by fine and mature fibres.

The Cotton Research Institute in Giza uses the new high volume instrument (HVI), to measures all seven critical parameters defined by the USDA, as well as a rapid HVI compatible maturity test by double compression air-flow (FMT3) which measures maturity and fineness. The present study adds the importance of fibre maturity to major fibre properties, especially in marketing.

Micronaire reading and crystallinity

Table 6 gives the micronaire values by FMT3 for the two levels of maturity in both cultivars. The "r" value of micronaire values with crystallinity percent is shown in Table 7.

A problem with micronaire reading is that a mature fine cotton may have the same wall cross-sectional (mtex) as an immature, coarse cotton. Alternatively, these two fibres may have

about the same surface area and thus the same micronaire value. Without an independent measure of maturity, the mature fine fibre will be confused with immature fibre and erroneously discounted for fineness and maturity.

The Micronaire instrument does not give true assessment of the thickness of the cell wall relative to the fibre diameter or perimeter any trash, dust or impurities in the raw cotton fibre may be give a fluctuation of Micronaire reading. Micronaire value from FMT is more accurate than from the Micronaire instrument, such that FMT samples are clean and randomised after the preparation of the sample for tests.

Mechanical properties and crystallinity

The relationship between fibre strength and crystallinity percent in the cotton cultivars under study are shown in Tables 6 and 7. Results showed a positive correlation between crystallinity percent with fibre bundle strength at 1/8 and 0 inch gauge, the "r" values were 0.83 and 0.89 respectively. Fibre strength was correlated with the molecular weight distribution of cellulose polymers (Timpa, 1989). The relationship between cellulose crystallinity and fibre strength has been examined in several instances (Duckett and Tripp, 1967).

Fibre strength translates directly into yarn strength. It is the most important criterion for rotor yarn spinning. Fibre length is important, a longer fibre adds little to the strength to coarser yarn counts, but in the fine count range, fibre length becomes increasingly essential.

Industry now needs stronger yarns, since fibre strength and fibre maturity (crystallinity) translate directly into the strength of rotor yarns (Deussen, 1992). Rotor spinning is very widespread. By one estimate, rotor spinning will be the dominant spinning technology in the US and the World by 2000, and ring spinning spindles will have diminished (Deussen, 1992).

Fine structural properties and crystallinity

The number of convolutions, number of reversals per cm ribbon width, actual angle convolution, as well as diameter and lumen width of the fibre after swelling with 18% caustic soda are shown in Table 6. The relationship between crystallinity percent with number of convolutions per cm and lumen width were 0.66 and -0.90, respectively (Table 7).

Giza 45, the ELS cultivar, and Giza 77, the new ELS, has the highest rate of convolutions and the lowest rate of reversals. Giza 45 has the lowest ribbon width and is the finest cultivar in Egypt (Nawar *et al.*, 1980).

The X-ray method of crystallinity determination assumes a two-phase model of microcrystallites embedded in an amorphous matrix of chain molecules. The two-phase model is a simplification of the fibre structure in view of the possibility of a range of intermediate degrees of order.

There was a highly significant positive correlation between maturity and number of convolutions per cm. (Nawar and Ghorab, 1989). Convolutions or twist are a consequence of the kidney shaped cellulose ribbon that constitutes the mature fibre. Convolutions are necessary for spinning cotton (Betrabet and Iyengar, 1964) and their number and distribution are two additional factors that affect fibre strength measurement (Triplette, 1992).

Conclusion

There was a close relationship between crystallinity percent with fibre maturity, fibre strength at 1/8 or 0 inch gauge, number of convolutions per cm. and lumen width. Giza 45 was the higher in waxy substances and fibre elongation %, it had the lower ribbon width and diameter after swelling. This study shows the importance of fibre maturity as a major fibre

property, especially in marketing.

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Table 1. Parameters of the resolved peak of ELS Egyptian cotton cultivars.

	Before extraction				After extraction	
Cultivar	Giza 45		Giza 45		Giza 77	
Maturity level	HM	LM	HM	LM	HM	LM
Parameters						
F_{101}	0.830	0.985	-0.366	0.978	0.662	0.635
A_{101}	12.5	12.5	12.3	12.2	11.6	12.5
W_{101}	2.42	3.00	1.63	2.20	1.86	2.60
P_{101}	15.2	15.1	15.2	15.0	15.2	15.3
F_{101}-	-0.212	-0.470	-0.300	-0.331	0.149	-0.523
A_{101}-	12.4	9.7	12.9	17.4	12.7	10.2
W_{101}-	2.40	2.44	2.32	1.88	1.99	1.83
P_{101}-	17.0	17.0	16.9	17.0	16.8	17.3
F_{002}	0.401	0.258	0.578	0.376	0.242	0.116
A_{002}	58.7	59.3	56.4	52.5	64.4	57.6
W_{002}	2.09	2.07	2.09	2.06	1.84	1.89
P_{002}	22.9	22.9	23.0	23.0	23.0	23.01
a	-0.132	-0.046	0.770	0.255	0.829	-0.217
b	0.030	0.003	0.020	0.081	0.031	0.108
c	0.119	0.012	0.006	0.005	0.004	0.004
d	-0.000298	0.002817	-0.000098	-0.000094	-0.000073	-0.000057

Table 2. Parameters of the resolved peaks of ELS Egyptian cotton cultivars with low maturity.

Parameters	Giza 45	Giza 77
F_{101}	0.769	0.911
A_{101}	14.2	13.4
W_{101}	2.16	2.37
P_{101}	15.01	15.3
F_{101-}	0.340	0.611
A_{101-}	15 76	9.94
W_{101-}	1.69	1.55
P_{101-}	17.0	17.3
F_{002}	0.547	0.223
A_{002}	49.2	55.8
W_{002}	1.96	1.84
P_{002}	23.0	23.0
F_{para}	0.466	-0.441
A_{para}	5.34	4.03
W_{para}	5 47	3.93
P_{para}	21.3	20.5
% area	9.07	8.46
a	0.108	-0. 570
b	0.117	0.220
c	0.00455	-0.00081
d	-0.000106	-0.000029

Table 3. The degree of crystallinity and the background scatter percentages in ELS Egyptian cotton cultivars.

Cultivar	Maturity level	Crystallinity percent	Background scatter percent
Before waxy substances extraction (BWSE)			
Giza 45	High	80.2	19.8
	Low	81.7	18.3
After waxy substances extraction (AWSE)			
Giza 45	High	80.5	19.6
	Low	67.3	32.7
Giza 77	High	81.6	18.4
	Low	-69.0	31.0

Table 4. The apparent crystallite sizes in nanometers

Cultivar	Maturity Level	L_{101}	L_{101}	L_{002}
	Before waxy substances extraction (BWSE)			
Giza 45	High	3.27	2.44	3.31
	Low	2.78	2.26	3.21
	After waxy substances extraction (AWSE)			
Giza 45	High	3.38	2.48	3.50
	Low	3.78	2.97	3.34
Giza 77	High	4.00	3.20	3.57
	Low	2.88	2.90	3.37

Table 5. The number of diffraction planes constructing the crystallites.

Cultivar	Maturity Level	L_{101}/d_{101}	L_{101}/d_{101}	L_{002}/d_{002}
	Before waxy substances extraction (BWSE)			
Giza 45	High	6	5	9
	Low	5	4	8
	After waxy substances extraction (AWSE)			
Giza 45	High	6	5	9
	Low	6	6	9
Giza 77	High	7	6	9
	Low	5	6	9

Table 6. Fine structural and physical fibre properties at two fibre maturity levels of Giza 45 and Giza 77.

Fibre properties	Giza 45		Giza 77	
	High maturity	Low maturity	High maturity	Low maturity
Mature percent	79	64	81	65
Micronaire reading	3.4	2.7	3.4	2.9
Fiber strength at 1/8 in (g tex)	36.2	33.7	35.4	31.5
Fiber strength at 0 in. (g tex)	55	49.6	55.3	53.1
Elongatlon %	5.8	5.8	5	4.7
Number of convolutions per cm	28.8	18.4	30.18	29.59
Number of reversals per cm.	10.45	9.5	6.5	6.27
Ribbon width (M)	15.1	15.5	18.7	18.0
Actual angle convolutlon (°)	25	24	32	29
Diameter (µm)	19.6	19.7	22.2	21.2
Lumen width (µm)	2	3.2	2.4	3.6
Waxy substances of the weight of moisture free %	0.53	0.71	0.49	0.51

Table 7. The correlation between crystallinity percent with fine structural and physical fibre properties.

Fibre properties	"r"
Mature percent	0.997
Micronaire reading	0.944
Fibre strength at 1/8 in.	0.828
Fibre strength at 0 in.	0.890
Number of convolutions per cm.	0.660
Lumen width (M)	-0.904

Figure 1. Diagrammatic representation of convolution angle.