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An Investigation to Identify Factors that Confer High Strength Properties to Normal Cottons: The Relationship Between Orientation Factors (X-Ray, Optical) and Tensile Strength

A.R. Kalyanaraman

The South India Textile Research Association, Coimbatore-641014, India

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SUMMARY

The correlation between the tensile strength of cotton fibres and orientation factors determined by X-ray and birefringence methods has been reported. For pooled data from four species of cotton the correlation coefficients between the strength and orientation factors was in the range 0·52–0·63. The orientation measured by birefringence increased more rapidly with tensile strength than the orientation determined by X-ray. By combining the information from the two techniques the contribution of the crystallite regions and the overall space and time orientation of the polysaccharide chains to tensile strength may be separated.

1. INTRODUCTION

The measurement of the mechanical properties of the fibrous polymers used in textile manufacture is a very important tool in the assessment of the quality of the material. These properties partly determine the wear life of the finished product. Any information available about the strength of cotton fibres, the most abundant naturally occurring and industrially important polysaccharide fibres, and how strength is dependent on other internal parameters will be of value in increasing the utilisation of cotton as a fabric-forming material. This paper relates the orientation parameters of cotton obtained by optical birefringence and X-ray methods to strength measurements made at '0' and $\frac{1}{8}$ in gauges using the stelometer. The object of the present work was to

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attempt to identify the structural features of the polymer responsible for the high strength in cottons.

Laboratory studies have shown that there are variations in the strengths of raw natural cottons. Thus, in breeding experiments small lots of cotton having unusually high strengths have occasionally been produced. The basic cause of this variation is not well understood. No information exists at present as to what morphological and structural characteristics distinguish these high-strength fibres from those of normal strength cottons of similar genetic type. It is only known that the degree of polymerization, spiral angle, fibrillar orientation, fibre birefringence and crystallite sizes are all correlated to some extent with fibre strength, but to date the mechanism of fibre rupture due to tensile stressing has not been linked with fibre morphology or fine structure (Betrabet & Iyengar, 1964; Eagle *et al.*, 1967).

Orientation of cellulose chains in cotton have been measured and correlated with several physical properties of the fibres by earlier workers (Meredith, 1946; Hertel & Craven, 1956). Based on orientation measurements this paper attempts to point out that the high strength properties of cotton depend both on the stacking of the crystallites and on the length-to-width ratio of the molecule itself.

2. EXPERIMENTAL

Strength–orientation measurements have been made on 22 cottons of American origin. In order to extend the information for the four different species some data have been included from another source: see Table 1 (Seshan, 1973). The tensile strengths for all the cottons have been estimated using the stelometer. Sample bundles of fairly uniform thickness have been prepared, and each reading is the mean of tests on ten bundles. For each cotton results have been obtained for '0' and $\frac{1}{8}$ in gauge lengths.

The X-ray orientation measurements have been made on the (002) intensity profile for the cottons indicated by a superscript *a* in Table 1 and on the (040) profile for the others. Since cellulose crystals are uniaxial, the orientation obtained from the (040) profile would be the same as the (002) profile. The orientation is estimated using the Hermans *et al.* (1946) procedure for paratropic reflection (002) and diatropic reflection (040).

TABLE 1

Family	*Cotton*	*Strength (mN/tex)*		*Strength retention*	*Orientation factor*	
		0 gauge	*⅛ in gauge*	*⅛ in gauge/ 0 gauge*	*X-ray* f_x	*Optical* f_o
Gossypium arboreum	Sanjay[a]	396·2	240·3	0·606	0·732	0·711
	CJ 73[a]	435·4	250·1	0·574	0·718	0·732
	Kovilpatti[a]	443·3	242·2	0·546	0·737	0·851
	Gaorani-6	466·8	228·5	0·489	–	0·768
G. barbadence	Sea Island[a]	370·7	223·6	0·794	0·671	0·701
	Sujatha	473·7	289·3	0·603	0·699	0·797
	Pima-1	454·1	298·1	0·657	0·762	0·833
	Pima-11	460·9	279·5	0·606	0·731	0·804
	Pima-20	404·1	292·3	0·711	0·747	0·838
	Pima-22	414·8	291·3	0·702	0·723	0·846
	Florence-2	405·0	225·6	0·557	0·771	0·791
	Florence-9	429·6	217·7	0·507	0·773	0·778
	Florence-18	372·7	220·7	0·592	0·758	0·791
	Florence-19	378·6	214·8	0·567	0·764	0·831
	Florence-21	393·3	236·4	0·601	0·766	0·844
G. hirsutum	PRS-72[a]	331·5	187·3	0·565	0·597	0·646
	MCU-1[a]	318·7	138·3	0·434	0·678	0·670
	Gujaret-67[a]	337·4	190·3	0·564	0·628	0·662
	Acala-1[a]	316·8	193·2	0·610	0·679	0·731
	Acala-4	367·8	192·2	0·523	0·741	0·675
	Acala-10	425·6	221·6	0·521	0·749	0·817
	Acala-14	409·0	226·5	0·554	0·750	0·802
	Acala-15	406·0	216·7	0·534	0·737	0·812
	Acala-16	397·2	221·6	0·558	0·744	0·813
	Deltapine-6	361·9	192·2	0·531	0·677	0·763
	Deltapine-8	341·3	183·4	0·537	0·672	0·732
G. herlaceum	Digvijay[a]	433·5	255·0	0·588	0·720	0·692
	V797	352·1	198·1	0·563	0·688	0·675
				Average 0·578		

[a] Data obtained by Seshan (1973).

2.1 Preparation of fibre bundle

A random sample of the cotton fibre under test was selected and combed using Fibrograph combs. Loose fibres were removed by repeated combing of the tufts. One end of the tuft was fixed between two strips of paper and the paper strips were glued together. After it had completely dried, the other end was recombed to remove the loose and short fibres and the free side of the bundle was pasted to paper strips as before to give a fibre bundle of 10 mm length. After drying, the paper strips were cut to size and clamped into the bundle holder (Kalyanaraman, 1978a, b, c, 1980).

The bundle holder was then transferred to the X-ray texture goniometer. By making a radial scan with the fibre bundle, the Bragg angle of reflection was accurately noted. The usual normal beam transmission technique is used for scanning equatorial reflections while symmetrical transmission technique is used for scanning meridional reflections. Any misalignment of the fibre bundle with reference to the zero position of the texture goniometer was corrected by noting the angular position of the texture goniometer corresponding to the centre of symmetry of the intensity distribution curve. In the present investigation such an error has never exceeded 1° of arc and in such cases this has been subsequently used as the zero position for azimuthal scanning. Using a point-to-point counting technique the azimuthal scanning of the reflection at its peak position was carried out with a pulse height discriminator and proportional counter. The azimuthal scanning was performed from 0 to 90° on both sides of the peak at equal intervals of 3° using a fixed time of 32 s, and the mean intensities were used in the calculations. The background noise was assumed to be linear and equal to the intensity at an azimuthal angle of 90°. The average orientation was reached after completing investigations on ten such bundles for each case. The experiments were carried out at a relative humidity of 65 ± 2% and temperature of 27 ± 1°C.

2.2 Birefringence measurements

The Becke line method has been used to determine the birefringence for each cotton. Fifteen to twenty slides with four fibres mounted on each have been examined. The fibres were dried first and then condi-

tioned at a standard relative humidity of 65%. Mixtures of α-bromonaphthalene and liquid paraffin in suitable proportions were used to measure the respective refractive indices. The values presented here are based on the weighted mean of the matching refractive indices in the directions parallel and perpendicular to the fibre and have been precisely estimated with an Abbé refractometer. The optical orientation factor is the ratio of the birefringence for a given cotton to the value of 0·0616 obtained for ramie, the best oriented of the naturally occurring cellulosic fibres. The results are presented in Table 1.

3. RESULTS AND DISCUSSION

Meredith (1951a, b) showed that the correlation of strength and orientation could be improved if cottons were divided into species. Hertel & Craven (1956) and Rubenfeld & Virgin (1957) established that the degree of correlation between strength and orientation depends upon the test length used. They also pointed out that the strength of cotton fibres is decided partly by orientation and partly by the presence of weaknesses in the fibre.

From this investigation and the observations of earlier workers a regression line has been fitted and Fig. 1 represents the variations of X-ray orientation and optical orientation with tensile strengths recorded on the stelometer. The optical orientation shows a steeper straight line and the scatter is less than the scatter in the X-ray orientation values. However, the scatter in both these orientation–strength plots is considerable, as with all correlations of structure factors and strength of fibres. The scatter reflects the complexity of the factors controlling the strength of cotton. The orientation measured by X-ray increases more slowly with tensile strength than the orientation measured optically. This trend is significant and it can be explained as follows.

X-ray diffraction depends not only upon the configuration of the electrons (chromophores) but also on how they are arranged in a lattice. Thus the X-ray orientation represents the average in both space and time of the configuration of the diffracting matter, whereas the optical orientation represents the time-average orientation of the carbohydrate chain.

From Fig. 1 it is clear that the two lines, optical orientation vs tensile strength and X-ray orientation vs tensile strength, intersect and as

orientation increases tensile strength improves. The behaviour of the optical orientation indicates that in high-strength cottons the cellulose molecular length-to-width ratio is higher than in the low-tensile-strength cottons. A similar view regarding microfibrils has been expressed by Kinsinger & Hock (1948). X-ray orientation also increases with tensile strength showing an improvement in crystallite orientation (Kalyanaraman, 1980). This means that in producing high-strength cottons the breeder has to look for cottons where both the length-to-width ratio of the polymer is high and the dispersion of the crystallite orientation is low. In addition, from Fig. 1 it is interesting to note that high-strength cottons have both high space and time order and when the strength is low space orientation is better than time orientation. This means that in low-strength cottons the crystallite stacking is better and this contributes more to the X-ray orientation than the optical orientation. It is also interesting to note that at $\frac{1}{8}$ in gauge length the tensile strength versus orientation factors relationship behaves in exactly the same way.

The regression line for '0' gauge strength vs X-ray orientation factor f_x and optical orientation factors f_o are

$$f_x = 0{\cdot}0058(\text{'0' gauge strength}) + 0{\cdot}4840 \quad (1)$$

$$f_o = 0{\cdot}0090(\text{'0' gauge strength}) + 0{\cdot}4048 \quad (2)$$

and the correlation coefficients are 0·5885 and 0·6116 respectively.

The corresponding regression lines for $\frac{1}{8}$ in gauge strength vs orientation are:

$$f_x = 0{\cdot}0051(\tfrac{1}{8}\text{ in gauge strength}) + 0{\cdot}6006 \quad (3)$$

$$f_o = 0{\cdot}0101\ (\tfrac{1}{8}\text{ in gauge strength}) + 0{\cdot}5294 \quad (4)$$

and the correlation coefficients are 0·4351 and 0·6110.

Equation (1) implies that when the X-ray orientation reaches 0·484, the gauge strength is zero. This signifies that at lower levels of orientation the linearity breaks down or the fibres with such orientation may be brittle with only bulk strength and no tensile strength at all. However, the above conclusions about non-linearity at low orientations have to be verified by studying cottons in this range.

It is very interesting to note that the slopes of the regression lines are almost the same for the two lines dealing with X-ray orientation factors (eqns (1) and (3)), and the same is the case for the optical orientation factors. Also, because of this, the angles between the two lines f_x versus

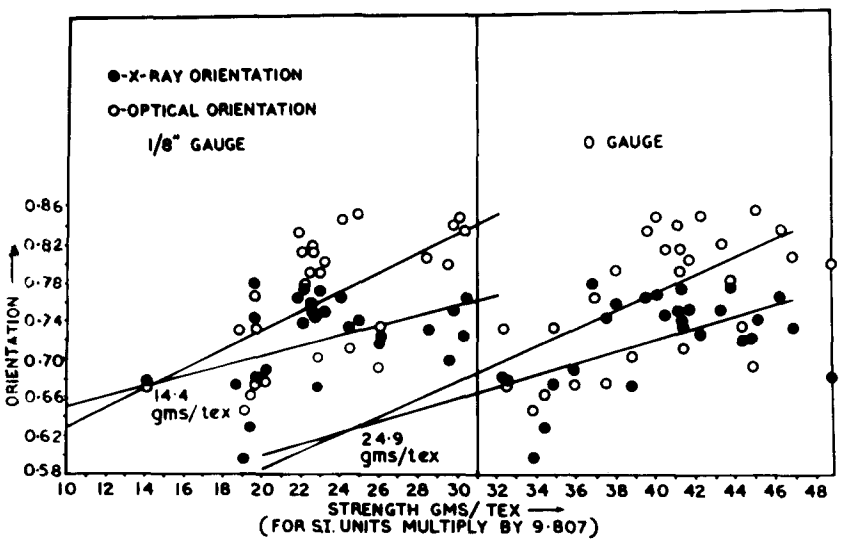

Fig. 1. Stelometer tensile strength plotted against orientation factors. The regression lines have been calculated assuming a linear relationship between orientation and tensile stress. As can be seen from Table 1, data from each species occupy a particular region in the figure.

'0' gauge strength and the equivalents at $\frac{1}{8}$ in gauge strength remain the same. This means that the internal order is not responsible for the fall in strength as gauge length increases. Further, it is clear that the fall in strength with the increase in gauge length is due to external factors like maturity, fineness of the fibre, etc., supporting the observation of Rubenfeld & Virgin (1957). From eqns (1), (2), (3) and (4), it is easy to evaluate the rate of increase in tensile strength with increase in orientation. Optical orientation as measured above represents time-average of the chromophores and X-ray orientation represents a time- and space-average. Thus this analysis suggests a method of quantifying the space-average contribution to orientation if proper convolution functions of the respective orientations are known.

The points of intersection of the lines have a special meaning. The ratio of the tensile strengths (Fig. 1) at the point of intersection of '0' gauge and $\frac{1}{8}$ in gauge length lines (244·3 mN/tex and 141·3 mN/tex, respectively), is exactly equal to the average strength retention value obtained in the Table, namely 0·578.

4. CONCLUSIONS

(i) For high-strength cottons, the molecules have high length-to-width ratios and well orientated crystallite regions.
(ii) The surface morphology and related factors are responsible for the variation of tensile parameters with gauge length.
(iii) This paper gives a method of estimating the space-average contribution to orientation. The optical orientation factors presented are given relative to ramie, which is assumed to be a highly oriented fibre with an optical orientation of unity. (These conclusions are to an extent dependent on this assumption.)

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