Crystallinity, Particle Size, and Mechanical Properties of Fibers in Some Egyptian and American Cotton Cultivars

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SYNOPSIS

The main objective of this study was to measure the crystallinity, particle size, and mechanical properties of fibers and to assess their relationship in 18 Egyptian and American cotton cultivars. Remarkable variations in crystallinity were found, ranging between 49 and 90%, whereas the range of differences in particle size was somewhat narrow, and so its effect on mechanical properties was not detectable. The cotton cultivars that have higher values of crystallinity are characterized by higher values of tenacity at either $\frac{1}{8}$ in. or zero gauge and stiffness and by reduced elongation. © 1993 John Wiley & Sons, Inc.

INTRODUCTION

It is well known that cotton fibers are composed mainly of cellulose, which belongs to the class of high molecular compounds. The elementry units of cellulose molecules are anhydro-D-glucose units. Beside cellulose, the fiber contains noncellulosic and admixture such as wax, ash, protein, and sugars. Knowledge of structural parameters such as crystallinity, particle size, orientation birefringence, and degree of polymerization is undoubtedly helpful in understanding and interpreting the differences in fiber properties and chemical reactivity.

Hermans[1] reported that 70% of cellulose in cotton fiber is crystalline, whereas the remainder is amorphous. Abdel-Aziz[2] reported that the cellulose crystallinity ratio is 63.3, 72.8, and 60.2% for Giza 70, Giza 75, and dendera cultivars, respectively. Hindeleh[3] found remarkable variations in crystallinity between cultivars, being 69% for Giza 45 and 53% for Ashmouni. He added that the cultivar that has a higher value of crystallinity is characterized by higher values of breaking tenacity and stiffness and by a lower value of breaking elongation. Eweid et al.[4] found that the crystallinity for Giza 67 and Giza 70 ranges between 61.5 and 75.25%. The difference between this value for Giza 70 and that determined by Abdel-Aziz[2] (63.3%) may be due to the two different methods used by the respective authors.

The measurements of crystallinity in cellulosic materials received much attention during the last three decades. Research workers suggested procedures that aimed at the differentiation between cellulose specimens for the purpose of obtaining quantitative ideas about the crystalline fraction or crystalline order (lattice order).

Shenouda and Viswanathan[5] estimated the crystallinity of a few native and chemically treated cotton specimens. They exhibit some variations in the values of crystallinity between the cultivars of native cotton.

The aim of this article was to measure the crystallinity, particle size, and mechanical properties of cotton and to assess their relationship.

MATERIALS AND METHODS

The present studies were carried on the following materials:

1. Established cultivars as well as new hybrids belonging to the two categories of the Egyptian cotton. These included
   (a) Six cultivars and hybrids belonging to the extralong staple category grown in Sakha.

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Journal of Applied Polymer Science, Vol. 50, 49–55 (1993)
 CCC 0021-8995/93/010049-07

(b) Six cultivars and hybrids belonging to the long staple category grown in Sakha.
(c) Two cultivars and hybrids belonging to the long staple category grown in central and upper Egypt.

2. Bima S 6 cultivar grown in Giza.
3. Three American cotton cultivars grown in Giza.

The lint samples used in this study were obtained from the Cotton Research Institute, Giza, during the 1990 season.

Studied Characters

1. Crystallinity

Several workers have used X-ray diffraction techniques for estimating the degree of crystallinity of cellulosic fibers: Hermans & Weidinger,[6] Segal et al.,[7] Wakelin et al.,[8] and Patil et al.[9] In all methods, the intensity of the scattered X-rays by the sample is measured as a function of the scattering angle.

In the method described by Wakelin et al.[8] and modified by Patil et al.,[9] the radial intensity distribution curve of the sample is synthesized by superposition of similar curves from purely standard crystalline and amorphous components. Although this method is too laborious, it is the most accurate one.[10] The method is based on the equation

$$f = (S - A)/(C - A)$$

where f is the degree of crystallinity, and S, C, and A are the X-ray scattering powers of the sample under test, crystalline, and amorphous standards, respectively, at a common value of 2θ. The scattering powers of the samples are measured in arbitrary units, at the same 2θ values. By plotting $(S - A)$ against $(C - A)$ at different values of 2θ, f is given by the slope of the line. Figure 1(a) and (b) shows examples. In this method, the sample irradiated and the intensity of the beam are kept constant from sample to sample. Therefore, the correction for air scattering and the Compton effect are constant and were eliminated from the terms $(S - A)$ and $(C - A)$. The Lorentz-polarization factor depends on the Bragg angle θ and the absorption factor depends on the angle θ, the shape, and the thickness of the specimen. The condition of the experiment mentioned above gives the difference of the scattering and, therefore, all these factors are eliminated.

The samples were prepared for X-ray examination by cutting the cotton fibers until they were reduced to pass a 20-mesh sieve. A specially designed stainless-steel mold was made to give circular specimens of 1.0 cm diameter. The pressed samples were placed in the specimen holder of a Diano X-ray diffractometer and the patterns were scanned over the range $2\theta = 4°$–$40°$ using CuKα radiation.

The 100% crystalline cellulose was prepared from ramie cellulose fibers and the 100% amorphous cellulose was prepared by ball-milling dry cotton for 20 h. The intensity data were normalized so that the areas under the intensity curves in the range $8° \leq 2\theta \leq 40°$ were the same for the standards as for the sample under test.

2. Crystallite Size

The crystallite size perpendicular to the (002) plane was determined using Scherrer's equation[11]:

$$\sigma = K \frac{\lambda}{\beta \cos \theta}$$

where K is the shape factor taken as 0.9. β is the true breadth (extrabreadth other than that due to experimental conditions). The experimental broadening was calculated using quartz powder as a standard. The broadening was measured by the method of integral breadth, given as

$$\beta = \int_{201}^{202} I(2\theta)d(2\theta)/I(2\theta_0)$$

i.e., the area under the diffraction peak divided by the peak maximum.

3. Mechanical Properties

All fiber tests were carried out in the Laboratories of the Cotton Research Institute, Agricultural Research Centre, Giza, at a constant relative humidity $65 \pm 2\%$ and temperature of $70 \pm 2°F$ ($21 \pm 1.1°C$):

1. Fiber tenacity and percentage of elongation were determined by a Stelometer instrument at zero and $\frac{1}{8}$ in. gauge length according to the standard method designated by ASTM D 1445-75.[12] The values of tenacity were given in terms of g/tex.
2. Fiber tenacity uniformity ratio was calculated as the ratio between tenacity at $\frac{1}{8}$ in. and tenacity at zero gauge length.
3. Stiffness of the fiber was calculated according to Grover and Hamby.[13]

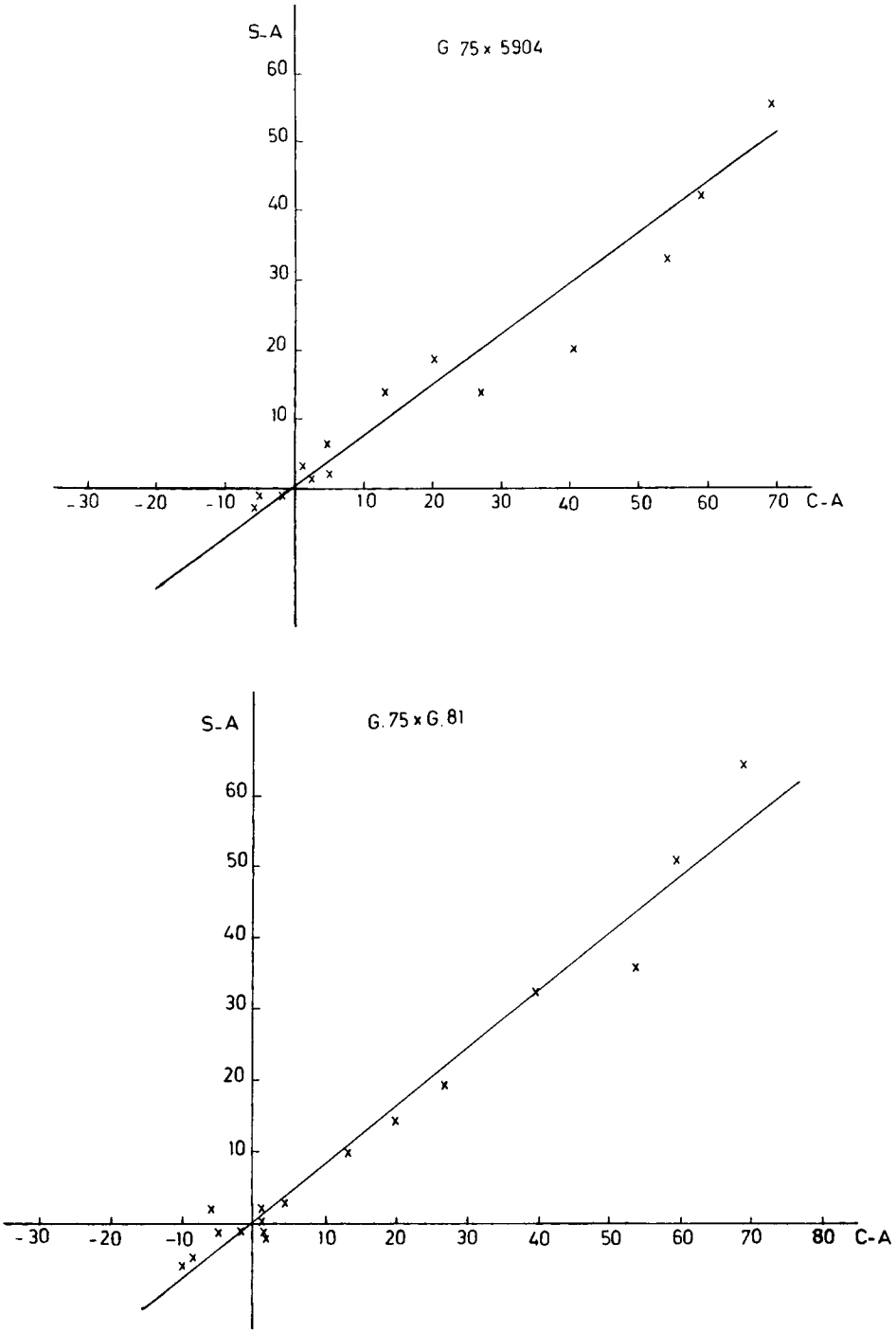

Figure 1 (a) The relation between $C - A$ and $S - A$ for Giza 75 × 5904. (b) The relation between $C - A$ and $S - A$ for Giza 75 × G81.

RESULTS AND DISCUSSION

The relevant specifications of the 18 cotton samples, i.e., crystallinity, particle size, and mechanical properties of the fibers, are listed in Table I.

Crystallinity

It is obvious that the crystallinity percentage ranges between 49 and 90%. The highest value was observed for the G.77 × G.45 hybrid (90%). On the contrary,

Table I Average Values of Structural Parameters and Mechanical Properties of Some Egyptian and American Cotton Cultivars

Cottons	Crystallinity (%)	Particle Size (Å)	Tenacity at $\frac{1}{8}$ in. g/tex	Tenacity at Zero g/tex	Tenacity Uniformity (%)	Elongation (%)	Stiffness (g/tex)
G[a].68 × C.B.58	86	45	34.80	48.14	72.29	6.10	570
G.76	84	46	37.32	51.68	72.21	6.00	622
G.70	85	49	32.18	51.92	61.98	6.02	534
G.77 × G.45	90	41	34.84	49.44	70.47	5.24	664
G.45	85	52	34.83	50.78	68.59	6.02	581
G.77	86	55	32.18	49.32	65.25	5.70	564
G.75 × 6022	88	49	28.48	42.83	66.50	6.11	466
G.75 × 5904	72	51	28.65	42.48	67.44	6.73	425
G.75 × G.81	81	49	29.71	48.50	61.26	6.10	487
G.67 × C.B.58	88	53	30.12	45.07	66.83	6.00	502
G.75	84	54	31.10	48.99	63.48	6.06	513
G.72 × Delciro	49	51	25.84	41.43	62.37	7.22	358
G.80	84	49	27.30	43.55	62.69	6.00	455
G.83	60	49	27.78	41.95	62.22	7.00	396
Bim S6	70	55	30.94	42.93	71.60	6.86	451
MchNair 220	58	48	26.33	41.84	62.93	7.10	371
Deltapine 70	87	50	28.16	46.02	61.20	6.10	462
Upland	85	55	28.20	42.83	65.84	6.05	466

G, Giza.

the G.72 × Delciro hybrid scored the lowest value of crystallinity (49%).

Particle Size

The true particle size ranges between 44 and 55 Å. It is clear that the range of differences between the 18 cultivars and hybrids is somewhat small, and, hence, the influence of particle size on the mechanical properties is not detectable in our present study.

Mechanical Properties of Fibers

Fiber tenacity at $\frac{1}{8}$ in. gauge ranged between 37.32 and 25.85 g/tex for Giza 76 and Giza 72 × Delciro hybrid, respectively. For the fiber tenacity at zero gauge, the highest value was recorded by Giza 70 (51.92 g/tex). In contrast, the lowest value was recorded by Giza 72 × Delciro hybrid. Tenacity uniformity ranged between 72.29% for the Giza 68 × C.B. 58 hybrid and 61.20% for Dettapine 70.

In regard to fiber elongation, the Giza 72 × Delciro hybrid was more elongated than were all the cultivars and hybrids (7.22%). In contrast, the lowest value in this respect was recorded by Giza 77 × Giza 45 hybrid, being 5.24%.

With respect to stiffness, the highest value of stiffness was scored by the Giza 77 × Giza 45 hybrid (664 g/tex). On the contrary, MchNair 220 scored the least, being 371 g/tex.

Relationship between the Crystallinity and Mechanical Properties of Fibers

The relationship between crystallinity and mechanical properties of fibers is shown in Table II and illustrated in Figures 2–5. The correlation coeffi-

Table II Simple Correlation Coefficient between Crystallinity and Mechanical Properties of Fibers

Characters		*r* Value
Crystallinity	× fibertenacity at $\frac{1}{8}$ in. gauge	0.54
	× fiber tenacity at zero gauge	0.60
	× tenacity uniformity	0.18 n.s.
	× fiber elongation	−0.045
	× fiber stiffness	0.74

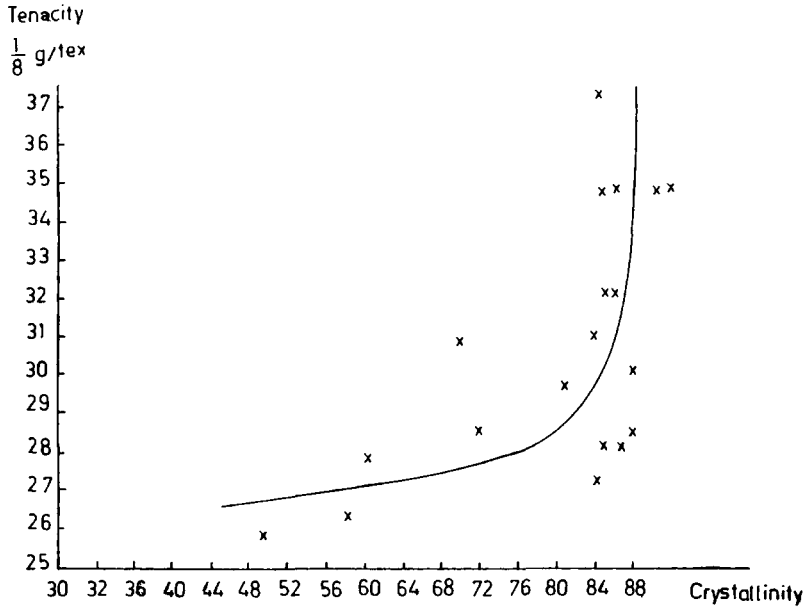

Figure 2 The relation between crystallinity and tenacity at $\frac{1}{8}$ g/tex.

cients between crystallinity and fiber tenacity at zero gauge and fiber stiffness (0.60 and 0.74, respectively) are positive and highly significant. However, the correlation coefficient between crystallinity and fiber tenacity at $\frac{1}{8}$ in. gauge is positive but not highly significant (0.54). The relationship between crystallinity and tenacity uniformity was positive but insignificant. In contrast, the correlation coefficient between crystallinity and fiber elongation was negative and significant. This means that cottons with high crystallinity will be expected to possess lower fiber extension at break.

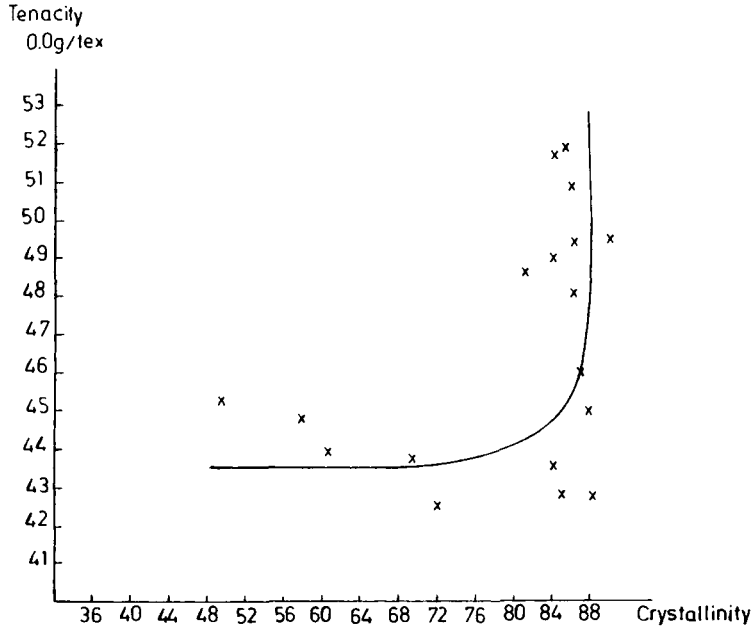

Figure 3 The relation between crystallinity and tenacity at 0.0 g/tex.

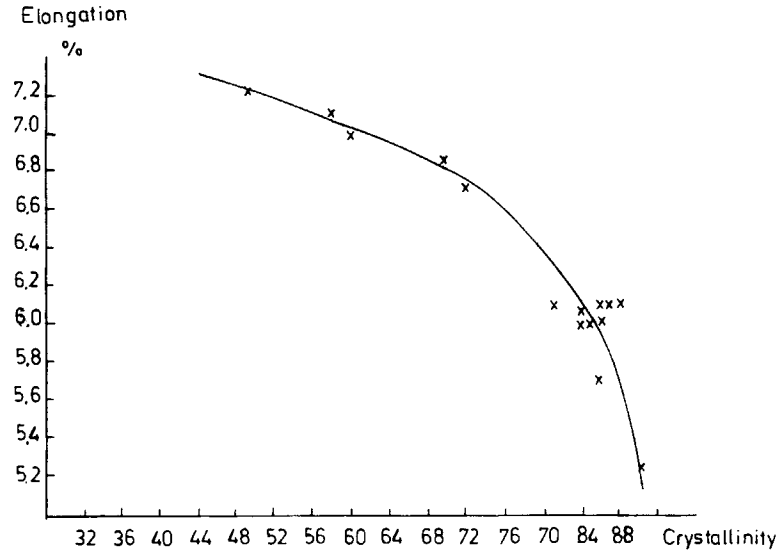

Figure 4 The relation between crystallinity and elongation.

It has been mentioned above that the effect of crystallinity on the fiber tenacity at zero gauge was higher (0.60) than its effect on fiber tenacity at $\frac{1}{8}$ in. gauge (0.54). This might be due to the large effect of weak links on tenacity when the bundle test length increases.

Assuming the aforementioned results, it might be concluded that the cotton cultivars that have

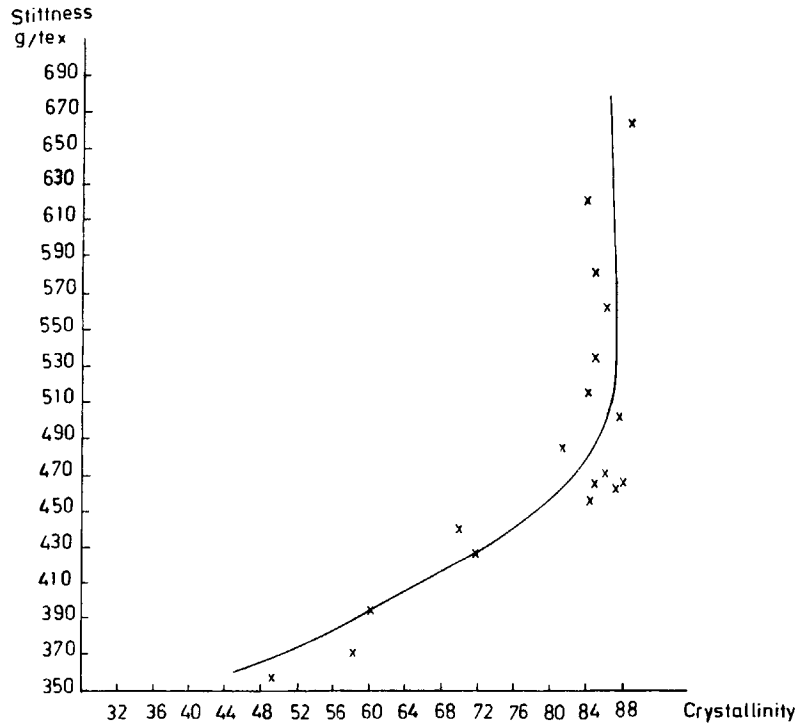

Figure 5 The relation between crystallinity and stiffness.

higher values of crystallinity are characterized by higher values of tenacity at either zero or $\frac{1}{8}$ in. gauge length and stiffness and by a lower value of elongation. This finding is in harmony with those obtained by Hindeleh and Johnson[14] and Hindeleh.[3]

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Received September 15, 1992
Accepted February 2, 1993