Characterization of Archaeological Cotton (*G. herbaceum*) Fibers from Yingpan

Qiuling Cao, Sukang Zhu, Ning Pan, Yuping Zhu, and Hengxian Tu

ABSTRACT

This paper examines the characteristics of some archaeological cotton fibers—*Gossypium herbaceum*—dating to A.D. 1161–1255, unearthed from Yingpan in the Xinjiang Autonomous Region, China. Three varieties of modern cotton including Jinta (*G. herbaceum*), SXY-1 (*G. arboreum*), and TM-1 (*G. hirsutum*) were selected for comparison with the unearthed fibers, and various approaches were used for the investigation, such as optical microscopy, scanning electron microscopy (SEM), infrared (IR) spectra, and x-ray diffraction (XRD). The archaeological fibers are found to share similar characteristics with the modern Jinta fibers, but are clearly different from the TM-1 fibers. Significant reduction in both tensile strength and breaking elongation is likely attributable to the degradation of the average degree of polymerization detected in the unearthed cotton fibers. It is suggested that using the spiral angle φ by XRD can potentially be a more effective and convenient way to distinguish among the diploid cotton and trapezoidal species from early times.

Introduction

About 150 km southeast of Yuli County in the Xinjiang Autonomous Region, the site Yingpan, where the cotton fibers were unearthed, is at a key point on the main route linking ancient China and neighboring countries to the west, as seen in Figure 1 (Institute of Archaeology of Xinjiang 2002). At the end of the 19th century and the beginning of the 20th century, the Swedes Hedin and Bergman, and Englishman Stein had visited the Yingpan site successively and excavated copper mirrors, glasses, silk, and other artifacts (Stein 1921; Hedin 1925; Baumer 2000). In 1995, archaeologists from the Institute of Archaeology of Xinjiang came to Yingpan and excavated about 120 ancient tombs already visited by tomb robbers and 32 intact tombs. Well-preserved mummies and other artifacts were unearthed, the excellent preservation due to the dry environment. The excavation was listed as one of the 10 most important archaeological discoveries in China in the year 1997 (Institute of Archaeology of Xinjiang 1999). Excavations at the tomb M29 produced about 100 g of seed cotton with boll shells. The fibers were later identified as a species of *Gossypium herbaceum* by the Cotton Research Institute, Chinese Academy of Agricultural Sciences, Anyang, China.

Subsequent radiocarbon dating conducted at Beijing University gave the cotton an age of 845 ± 40 B.P. Table 1 lists the results of the radiocarbon analyses. The Calibration program OxCAL and correction curve IntCal04 were used.

Cotton is a seed hair fiber of *Gossypium* of the family Malvaceae. *G. arboreum* and *G. herbaceum* both belong to the cultivated diploid species, whereas *G. hirsutum* and *G. barbadense* are cultivated tetraploid species (Brubaker et al. 1999). According to Chinese historical literature, *G. arboreum* was widely cultivated in ancient times throughout China, while *G. herbaceum* was only planted in the ancient Xinjiang region. *G. hirsutum*, however, replaced both *G. arboreum* and *G. herbaceum* soon after it was introduced to China as early as the 19th century, spreading throughout cotton growing regions (Gillham et al. 1995).

This study examines the characteristics of the unearthed fibers by using optical microscopy, scanning electron microscopy (SEM), infrared (IR) spectra, and x-ray diffraction (XRD), and establishes a more-detailed data record for the ancient cotton fibers. Also, in order to have a better understanding of the archaeological cotton fibers, samples of three modern cotton species, Jinta (*G. herbaceum*), SXY-1 (*G. arboreum*), and TM-1 (*G. hirsutum*) were used as a comparison.

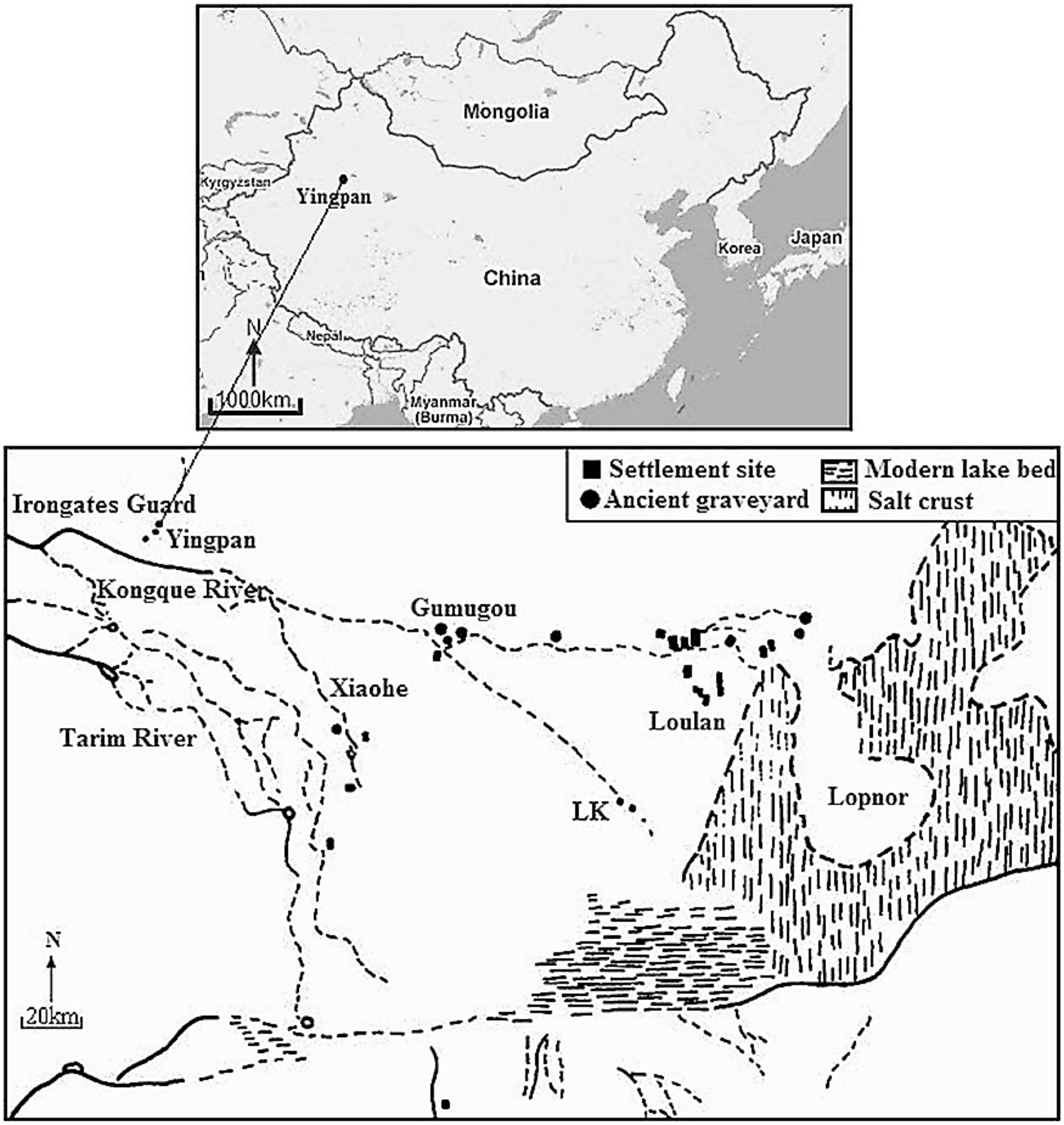

Figure 1. Location of the Yingpan site. (Drawing by authors, 2008).

Table 1. Radiocarbon Analysis.

Lab no.	Radiocarbon age (B.P.)	Calendar year	
		1 sigma SD (68.2%)	2 sigma SD (95.4%)
BA06819	845 ± 40	A.D. 1161 (68.2%) A.D. 1255	A.D. 1040 (9.8%) A.D. 1090 A.D. 1120 (85.6%) A.D. 1270

The Analysis

Samples of the unearthed cotton fibers were provided by the Institute of Archaeology of Xinjiang, and the samples of contemporary cotton fibers were provided by the Chinese Cotton Germplasm Resource Center, Anyang, China.

Length, Fineness, Ribbon Width, Convolutions, and Tensile Measurements

One hundred single fibers each from the unearthed sample, and the modern Jinta, SXY-1 and TM-1 samples were selected for testing. Fibers were placed on a wet-pile blackboard with forceps, and were straightened gently. The length of each fiber was measured using a ruler. Next, according to the ASTM D1577 Standard Test Method for Linear Density of Textile Fibers, Option C-Vibroscope, each sample's fineness was measured using an XD-1 Fiber Fineness Tester. Ribbon width and convolution were then determined using an Olympus CH-2 video microscope. Afterwards, based on the ASTM D3822 Standard Test Method for Tensile Properties of Single Textile Fibers, the tensile properties of the samples were tested using an XQ-1 Tensile Tester at a gauge of 10 mm and drawing speed of 10 mm/min.

Scanning Electron Microscopy Tests

The shapes of the unearthed fibers and the Jinta fibers were investigated in more detail using an Hitachi JSM-5600LV scanning electron microscope with an accelerating voltage of 15 kV. Specimens were coated with platinum to avoid charging effects.

Determination of the Degree of Polymerization

According to the ASTM D1795 Standard Test Method for Intrinsic Viscosity of Cellulose, the average degree of polymerization of both the unearthed fibers and the Jinta fibers was determined by measuring the polymer viscosity, performed in cuprammonium solution at 25 ± 1°C with an Ubbelohde viscometer (Jin 2000).

Fiber Density

According to the ASTM D1505 Standard Test Method for Density of Plastics by the Density-Gradient Technique, normal heptane-carbon tetrachloride was selected as the liquid system for treatment, and five glass floats were used to calibrate the density-gradient tube. The densities of the unearthed fibers and the Jinta fibers were thus tested by the density-gradient method. The test specimens were pretreated as follows: soil particles adhering to the fibers were removed by immersing the fibers in distilled water for two hours; the fibers were then air-dried and knotted into fiber balls; cotton wax was removed by treatment in ether for two hours; and the fiber balls were then air-dried at room temperature.

Infrared Spectra

The IR spectra were obtained with a Nexus 670 spectrophotometer using the attenuated total reflection (ATR) technique (ZnSe crystal), and recorded at 4 cm^{-1} resolution. Sixty-four scans were taken per sample. Nelson and O'Connor's (1964a) IR crystallinity ratio *K* was calculated using the following formula:

$$K = a_{1372} / a_{2900}$$

Where a_{1372} is the intensity of absorption at 1372 cm^{-1}, and a_{2900} the absorption at 2900 cm^{-1}. According to Nelson and O'Connor's (1964a; 1964b) work on the structure of cellulose, the a_{1372} band is associated with the C–H bending mode and affected by changes in crystallinity, while the a_{2900} band is associated with the C–H stretching vibration and unaffected by changes in crystallinity.

X-Ray Diffraction Tests

Wide-angle x-ray diffraction measurements of samples were carried out with a D/max-2550PC diffractometer of CuKα radiation operated at 40 kV, 300 mA with the wavelength $\lambda = 1.54056$ Å. The diffraction patterns of all samples were recorded over the 7°–50° 2θ range continuously at a scan rate of 10°/min. In preparation for the x-ray goniometer, the fiber samples, before being mounted on a sample holder, were cut with scissors into lengths of approximately 0.5 mm to diminish the possibility of preferred orientation effects. The spectra were peak resolved by the software with the apparatus using the pseudo-Voigt method. The formula for calculating the crystallinity is the following:

$$X_c = \frac{I_c}{I_c + I_a} \times 100\%$$

where X_c is the crystallinity; I_c is the sum of the crystal peaks' diffraction intensity, and I_a the diffraction of the amorphous peak. The crystallite size was computed from the half width of the line profiles using the Scherrer equation.

The samples were aligned and installed in the sample holder for testing. The (002) arc was analyzed for studying the cotton-fiber orientation. According to Deluca and Orr's method, both the crystallite orientation and spiral angle can be directly determined from the x-ray tracing of the (002) diffraction arcs from the combed bundles of cotton fibers (Deluca and Orr 1961). Furthermore, if the distribution of crystallites is assumed to be Gaussian, the experimental arc can be generated theoretically. A separated (002) result is shown in Figure 2. The peak separation was completed using least-square methodologies. The

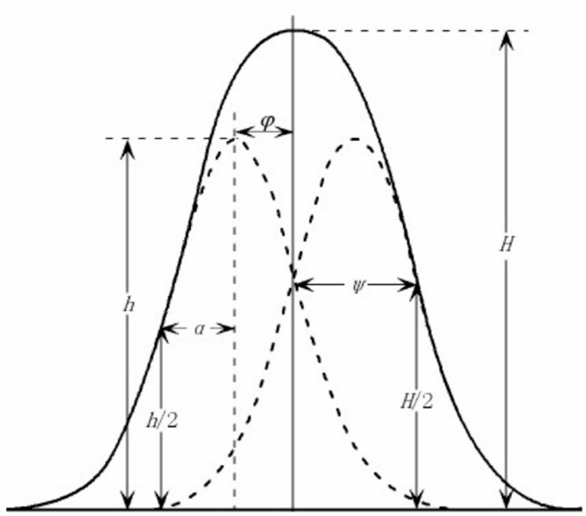

Figure 2. Sketch of a separated (002) result. (Drawing by authors, 2007).

50% x-ray angle ψ characterizes the total orientation of cellulose macromolecules; the spiral angle φ indicates the angle of the fibrils relative to the fiber axis; and the crystallite orientation angle α represents the discrete degree of orientation of the b-axis in the microcrystalline structure relative to the fiber axis (Tao et al. 1996).

Results and Discussion

Comparison of Fiber Characteristics

The measured data of fiber length, fineness, ribbon width, convolutions, strength, and elongation are shown in Table 2. Those data reveal the length of the unearthed fibers is 18.3 mm, similar to the 18.7 mm of the Jinta fibers, but the length of the TM-1 fibers is 28.9 mm, significantly longer than all other species. The ribbon width of the unearthed fibers is 22.3 μm, slightly wider than the Jinta (21.4) and slightly narrower than the SXY-1 (23.8), but much wider than the TM-1 (17.3). The fineness of the unearthed fiber, 0.27 tex, is greater than that of all the other three fibers (0.23, 0.26, and 0.16); this may well be because of possible remaining soil on the unearthed fibers, leading to the increase of their dimensions. The convolutions are characterized by the number of 180° reverses per centimeter in length. The unearthed fibers convolute about 53 times per centimeter, slightly fewer than those of the Jinta and the SXY-1 fibers, and the TM-1 sample exhibits the highest number of convolutions.

Moreover, there seems to be some correlation between fiber length, fineness, and convolutions: shorter fibers, such as the Jinta and the SXY-1 fibers, are thicker and have fewer convolutions, while TM-1 fibers are longer and finer, and have more convolutions. So, the fibers of *G. hirsutum* (TM-1) are more suitable for machine processing, and production quality is better. This may be the main reason *G. hirsutum* rapidly replaced both *G. herbaceum* and *G. arboreum* and became the prevalent species planted.

The fiber strength and elongation of the unearthed fibers are less than those of the modern fibers. A certain loss of original physical properties is expected with all unearthed ancient textiles. The fiber strength of some *G. arboreum* cotton samples unearthed from Mohenjo-daro and dated to 2600–2000 B.C. (Fuller 2008) was reported

Table 2. The Length, Fineness, Ribbon Width, Convolutions, Strength, and Elongation of Fibers.

Sample	Length (mm)	Fineness (tex)	Ribbon Width (µm)	Convolutions (cm^{-1})	Strength (cN)	Elongation (%)
Unearthed	18.3	0.27	22.3	53	3.02	4.03
Jinta	18.7	0.23	21.4	59	5.09	5.32
SXY-1	23.0	0.26	23.8	58	6.17	5.73
TM-1	28.9	0.16	17.3	77	3.58	5.92

to be about 2.69 cN (Gulati and Turner 1929), indicating that the fibers were obviously deteriorated. The tensile properties of modern cotton fibers depend mainly on their supermolecular structure (Boylston and Hebert 1983), thus indicating that time and archaeological context have an adverse impact on fibers' molecular morphologies.

Morphology Analysis by SEM

Figures 3 and 4 show the SEM images of both the unearthed and Jinta fibers under different magnifications. The SEM analysis reveals that the unearthed fibers and the Jinta fibers have typical cotton morphological features in common, such as the ribbon shape rolled in a helicoidal manner around the axis, and more-or-less bean-like cross sections. Figure 3 indeed shows some residual soil particles attached to the unearthed fiber surface. Figure 4*a* exhibits a surface of roughly parallel ridges and grooves spiraling around the fiber at an acute angle to its axis, believed to be a reflection of the spiral fibrillar structure of the cellulose beneath the primary wall of the cotton fiber (Shenouda 1979). The ridges and grooves cannot be seen from the unearthed fiber, however, either having disappeared due to aging or perhaps still covered by the soil. If clearer images are desirable, one could clean the sample more thoroughly or use image processing tools (Kearns et al. 2003).

Degree of Polymerization Analysis

The average degree of polymerization of the unearthed and Jinta fibers is 2586 and 3300 respectively. This may indicate the degree of polymerization of the cellulose decay after the cotton fibers were buried in soil for a long period of time, which accounts for the reduction in both strength and elongation of the fibers shown above.

Density Analysis

As seen in Table 3, the experimental results on both fibers are close to that of published data reported by (Morton and Hearle 1993). The average density of the unearthed fibers is 1.546 g/cm^3, slightly higher than the 1.541 of the Jinta fibers, and this tiny difference could be caused by experimental errors or the presence of heavier soil remaining on the fibers. As the data in Table 4 in the next section indicates, however, the crystallinity of the unearthed fibers is indeed somewhat higher than that of the Jinta fibers.

Infrared Spectra Analysis

The IR spectra of both the unearthed fibers and the Jinta fibers are provided in Figure 5. The IR spectra of the samples are different from that described by Nelson and O'Conner (1964a). This is likely due to differences in sample preparation methods: this study adopted the ATR technique, whereas Nelson and O'Connor used the potassium bromide pellet technique. The ATR technique is suitable for the study of archaeological materials since it analyzes the surface composition without causing destruction, and the resultant fingerprint characteristics of the spectra are clearer and easier to investigate. The *K* values of the unearthed fibers and the Jinta fibers are 4.4 and 3.7 respectively, clearly indicating that the IR crystallinity ratio of the unearthed fibers is higher than that of Jinta samples; the higher the IR crystallinity ratio of a cellulose, the stronger the intermolecular hydrogen bonds, and the neater the packing arrangement of the molecules.

Still, it is seen from Figure 5 that the IR spectra of the unearthed fibers are similar to the spectra of the modern Jinta fibers. Yet the peak due to –OH stretching vibration in

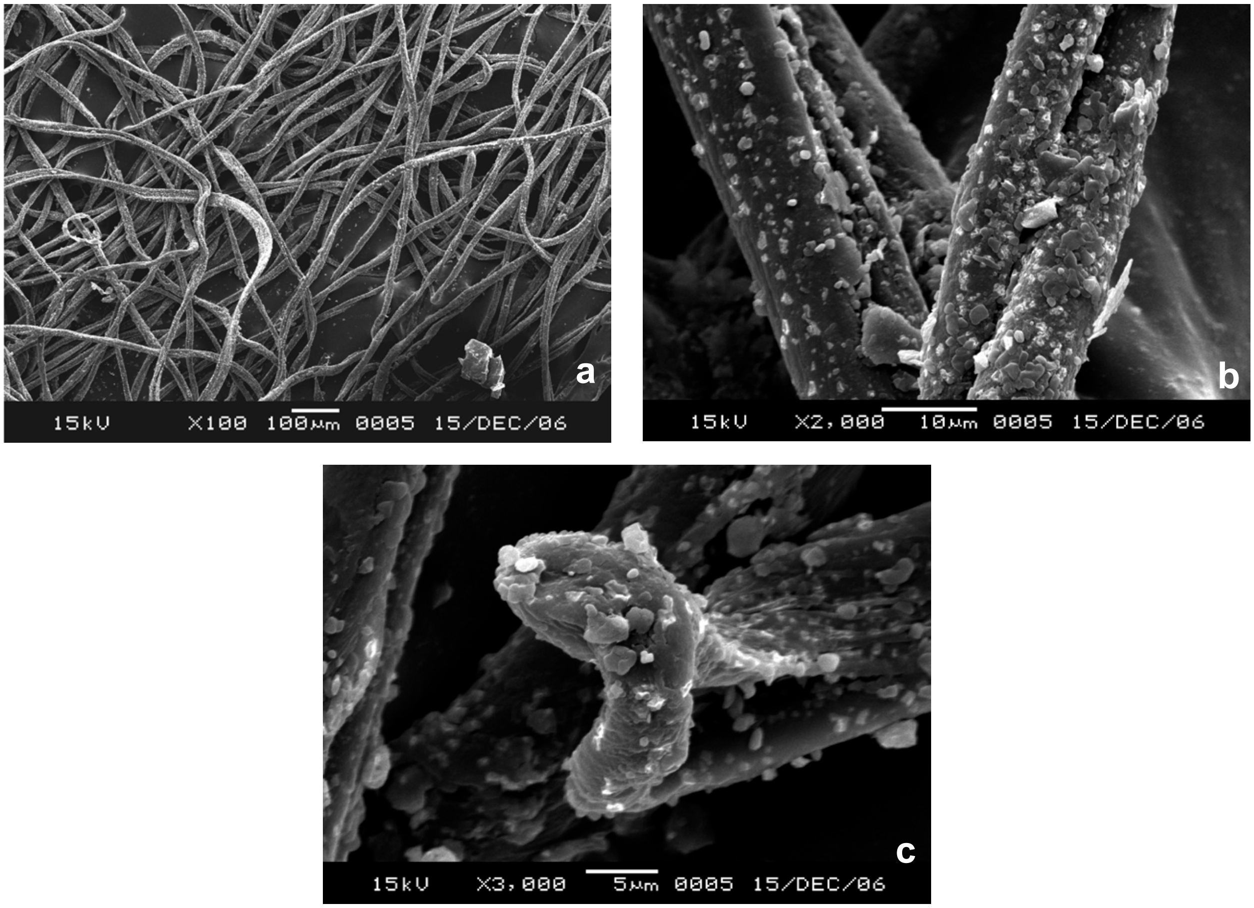

Figure 3. SEM images showing: *(a)* the longitudinal morphology of the unearthed fibers under 100× magnification; *(b)* the longitudinal morphology of the unearthed fibers under 2000× magnification; *(c)* the cross-sectional morphology of the unearthed fibers under 3000× magnification. (Photos by authors, 2006).

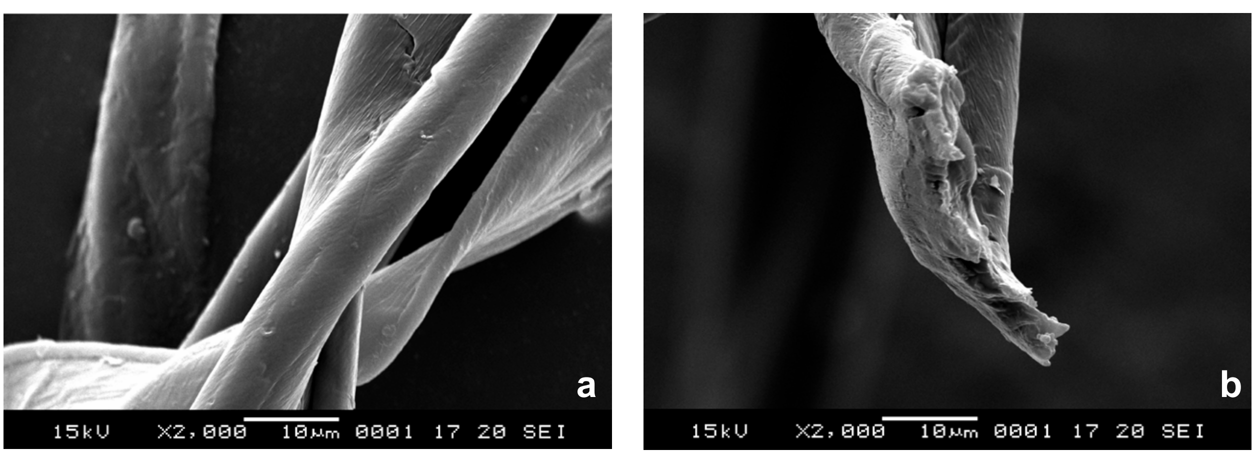

Figure 4. SEM images showing: *(a)* the longitudinal morphology of Jinta fibers under 2000× magnification; *(b)* the cross-sectional morphology of Jinta fibers under 2000× magnification. (Photos by authors, 2006).

Table 3. The Density of Fibers, g/cm^3.

Sample	1	2	3	4	5	Average
Unearthed	1.543	1.546	1.544	1.549	1.546	1.546
Jinta	1.540	1.541	1.540	1.541	1.542	1.541

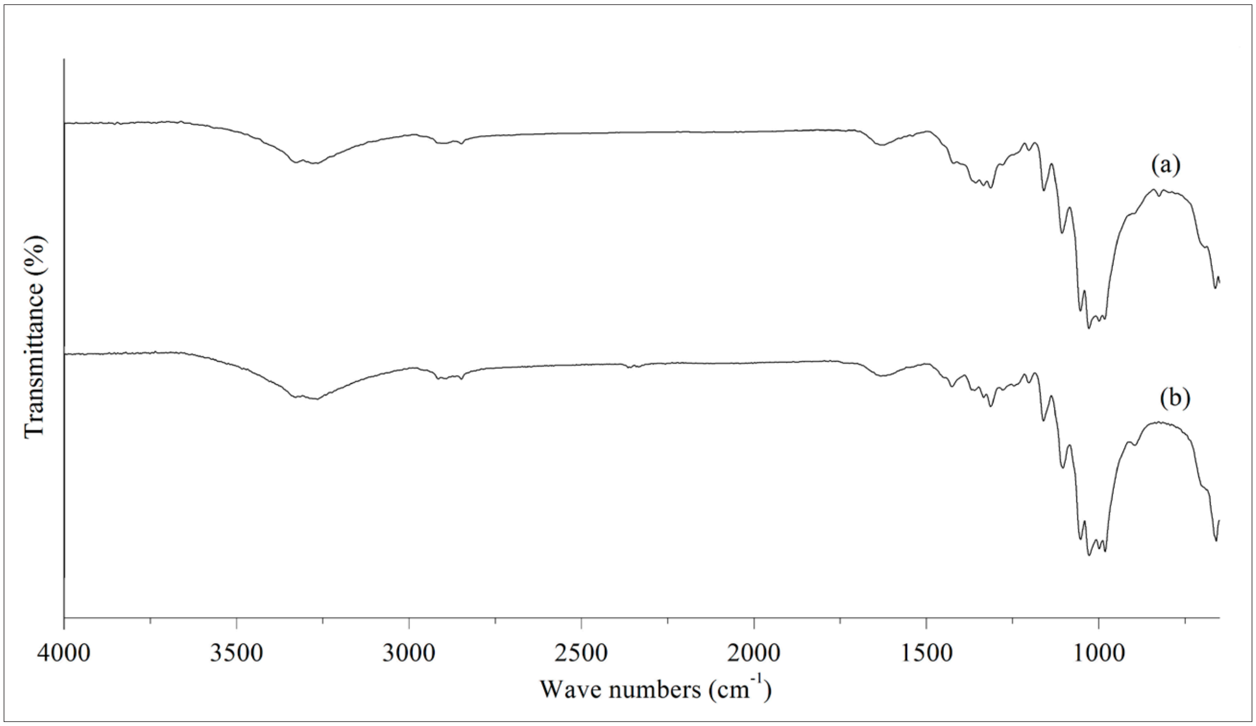

Figure 5. IR spectra of samples: (*a*) Unearthed, (*b*) Jinta. (Drawing by authors, 2007).

the absorption range of 3200 to 3400 cm^{-1}, the major characteristic absorption peak for cellulose fibers, appears to be more shallow and narrow. Nearly identical tiny peaks at 2895 cm^{-1} associated with C–H stretching, and 2849 cm^{-1} associated with –CH_2 symmetrical stretching vibration are seen on both curves; the physically absorbed water molecule shows a characteristic peak at 1630 cm^{-1} (Parmar and Giri 2001); peaks at 1357 are due to C–H bending, 1335 to O–H in-plane bending, and 1315 cm^{-1} to –CH_2 wagging; the band at 1160 cm^{-1} is associated with C–O stretching or O–H bending of the C–OH group (O'Conner et al. 1957); peaks near 1100 cm^{-1} are attributed to the hydrogen bonding on the skeletal vibrations which involve stretching of the C–O bond; the band at 893 cm^{-1}, assigned to motions of atoms attached to C_1, reflects the changes in molecular conformation due to rotation about the C_1–O–C_4 (glucosidic) linkage (Nelson and O'Conner 1964b). A new peak, however, appears at 826 cm^{-1} in the unearthed cotton and is likely attributable to inorganic particles in the soil.

X-Ray Diffraction Analysis

The XRD spectra of the samples are shown in Figure 6. All the XRD intensity profiles of the fibers show a well-resolved spectrum of Cellulose I, with three characteristic reflections at (101), i.e., $2\theta = 14.7°$; $(10\bar{1})$, 16.6°; and (002), 22.7°. The first two are at medium intensity and convoluted with each other, yet the third is at a very sharp, strong intensity. This indicates that the basic internal morphology of the unearthed fibers is not altered after nearly a millennium of burial underground. The smaller peak at (040) or $2\theta = 34.4°$ is another feature shared by all the cotton fibers tested. Diffraction peaks of the unearthed fiber (Figure 6*a*) at 26.7°, 31.7°, and 45.5° are caused by the

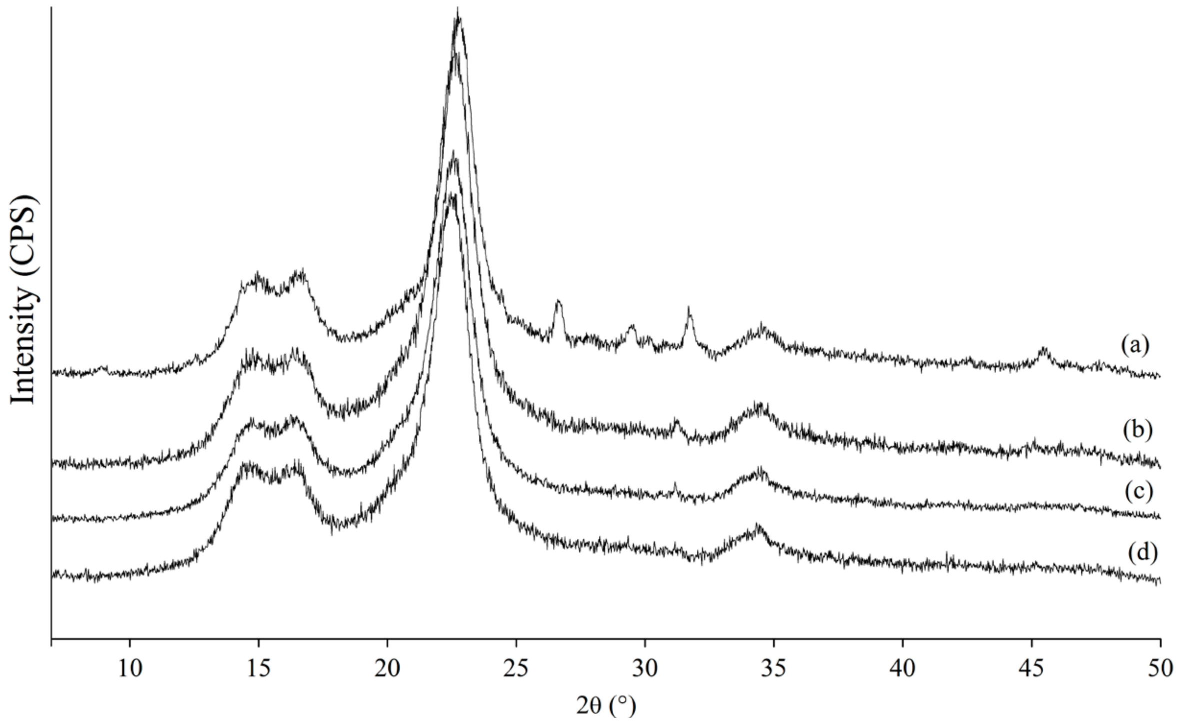

Figure 6. XRD spectra of samples: (*a*) Unearthed, (*b*) Jinta, (*c*) SXY-1, (*d*) TM-1. (Drawing by authors, 2007).

silicon dioxide and sodium chloride from the soil particles adhering to the unearthed fiber. The crystallinity, crystallite size, and orientation parameters of the four sample types are given in Table 4.

As those data reveal, the values for the two species SXY-1 (78.0%) and TM-1 (73.8%) are higher. Since they are of the same species, the crystallinity of the unearthed fiber is 71.7%, comparable to 70.7% of the Jinta sample. This indicates that the crystallinity of the ancient fibers remains largely unchanged. In terms of the crystalline scale, at plane (101) crystallite-size ranking of the samples are unearthed fibers = TM-1 > Jinta > SXY-1, and at plane ($10\bar{1}$) they all share a similar crystallite size, except for TM-1, which has a lower value. The crystallite size at plane (002) characterizes the crystallites at the horizontal direction, i.e., along the fiber axis, and the value of the unearthed fibers is 60 Å, significantly larger than those of the modern fibers. It may be a little premature to say that the cotton fibers from antiquity had some structural differences from the Jinta fibers today. It is safe, though, to conclude that the dry conditions in Xinjiang have preserved the fibers well, even over such a long period of time.

Further examination of other angular parameters confirms that both the cultivated diploid species such

Table 4. The Crystallinity, Crystallite Size, and Orientation Parameters of Samples.

Sample	Orientation Parameters (°)			Crystallite Size (Å)			Crystallite (%)
	50% x-ray angle ψ	Spiral angle φ	Crystallinity orientation angle α	101	$10\bar{1}$	002	
Unearthed	22.7	9.8	20.1	39	56	60	71.7
Jinta	22.7	9.0	21.9	35	56	48	70.3
SXY-1	23.7	9.7	18.3	33	57	49	78.0
TM-1	31.3	15.2	20.5	39	51	52	73.8

as *G. arboreum* (SXY-1) and *G. herbaceum* (unearthed samples and modern Jinta) possess the smallest spiral angle φ among all the cultivar cotton species (Moharir and Vijayraghavan 1993; Tao et al. 1996), and 50% x-ray angles smaller than those of the tetraploid species like *G. hirsutum* (TM-1). In other words, this provides another piece of evidence that the unearthed fibers belong to the cultivated diploid and not the tetraploid species. The crystallite orientation angles α for all the samples are very close. In archaeological discoveries, ancient organic materials such as textiles are difficult to preserve. Accordingly, it is difficult to identify the species from the appearance of single fibers (Moharir 1980; Good 2001; Moulherat 2002). As cotton fibers are single-cells composed mainly of cellulose, it is also difficult to conduct DNA analysis, especially on ancient samples with certain degradation. The use of the spiral angle φ by XRD, however, can potentially serve as a more effective and convenient way to distinguish between diploid and tetraploid cotton species from early times.

Conclusions

This study examined fibers (*G. herbaceum*) unearthed from Yingpan in the Xinjiang Autonomous Region, China, dated to A.D. 1161–1252, comparing them with fibers from some modern cotton species. The unearthed fibers have an average length of 18.3 mm, fineness of 0.27 tex, and convolutions of approximately 53 turns per centimeter. All unearthed samples are similar to the modern Jinta (*G. herbaceum*) fibers but distinctively different from the TM-1 (*G. hirsutum*) fibers. The reduced average degree of polymerization caused by long-time burial is considered the main cause for the decrease in both strength and elongation of the unearthed cotton fibers. Because tensile fracture in a cotton fiber is mainly due to damage in its amorphous regions, the decline of the average degree of polymerization results in the reduction of both tensile strength and breaking elongation of the unearthed fiber. SEM analysis shows that the unearthed fibers remain more ribbon-like, rolled in a helicoidal manner around the axis, and with bean-like cross sections.

The IR crystallinity ratio of the unearthed fibers is higher than that of the Jinta fibers. The XRD intensity profile of the unearthed fibers shows the typical spectrum of Cellulose I, with the three characteristic reflections at (101), ($10\bar{1}$), and (002), as well as the peaks caused by the silicon dioxide and sodium chloride in the residual soil associated with the unearthed fibers. Both the crystallinity and the crystalline sizes of the unearthed fibers are no less than those of the modern fibers, suggesting the dry conditions have otherwise largely protected the fiber morphology.

Further examination of other angular parameters confirms that both the cultivated diploid species such as *G. arboreum* (SXY-1) and *G. herbaceum* (the unearthed and Jinta) possess the smallest spiral angle φ among all the cultivar cotton species, as well as 50% x-ray angles smaller than those of tetraploid species like *G. hirsutum* (TM-1). In other words, this provides another piece of evidence that the unearthed fibers do belong to the cultivated diploid, and not the tetraploid species. Furthermore it is thus suggested that the spiral angle φ by XRD is a more effective way to distinguish diploid cotton fiber from that of the tetraploid species.

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Qiuling Cao
College of Textiles
Donghua University
Shanghai 201620, China

Sukang Zhu
College of Textiles
Donghua University
Shanghai 201620, China

Ning Pan
Division of Textiles and Clothing
Biological and Agricultural Engineering Department
University of California
Davis, CA 95616

Yuping Zhu
Research Center of Analysis and Measurement
Donghua University
Shanghai 201620, China

Hengxian Tu
College of Textiles
Donghua University
Shanghai 201620, China