(REFEREED RESEARCH)

INVESTIGATION OF THE INTERNAL STRUCTURE OF REGENERATED BAMBOO FIBER

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ABSTRACT

Regenerated cellulose fibers are gaining importance in textile industry with the increase in human population and the advances in technology. Bamboo fiber is a cellulose fiber with antibacterial and easy dyeability properties and a soft feeling and its use is increasing as the healthy living trend gains momentum and impacts the textile industry. To have an in-depth knowledge of bamboo fiber, the internal structure, degree of polymerization, percent crystallinity ratio and particle size of bamboo fibers were examined and compared with those of 100% cotton fibers. According to the results obtained, the differences in mechanical properties of fibers, were found to be closely related to the variations in their internal structures.

Key Words: Regenerated bamboo fiber, % crystallinity, Degree of polymerization, Viscosity, SEM.

ÖZET

İnsan nüfusunun hızla artışı ve teknolojide meydana gelen gelişmeler sonucunda tekstil endüstrisinde rejenere selülozik lifler giderek önem kazanmaktadır. Bambu elyaf da anti bakteriyel, kolay boyanabilirlik, yumuşak tutum gibi özelliklere sahip olan rejenere selülozik bir liftir ve hızla yayılmakta olan sağlıklı yaşam trendinin tekstile de yansımasıyla kullanımı giderek artmaktadır. Bambu elyaf hakkında derinlemesine bilgi sahibi olunması amacıyla, liflerin iç yapısı, polimerizasyon derecesi, % kristalinite oranı ve tanecik boyutu incelenmiş, elde edilen sonuçlar % 100 pamuk lifleri ile karşılaştırılmıştır. Liflerin farklı mekanik özelliklere sahip olmalarının, iç yapılarındaki farklılıklardan kaynaklandığı tespit edilmiştir.

Anahtar Kelimeler: Rejenere bambu lifi, % kristalinite, Polimerizasyon derecesi, Viskozite, SEM.

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1. INTRODUCTION

Fibers used in textile industry are large molecule polymers consisting of long chains. The molar mass of polymers is a major criteria which affects their properties and defines their direct use applications. For polymers, a higher degree of polymerization (DP) and therefore molar mass indicates longer chains. Lengthening of chains increases chain entanglement thereby reducing their solubility (1). In addition to chain length determined by DP, the crystalline structure and size of the molecule is also important.

Different mechanical properties of individual cellulose fibers arise from their different degrees of polymerization and crystal sizes.

The regions where cellulose macro-molecules are ordered are called crystalline regions while the regions they are non-ordered are called amorphous regions. The abundance of crystalline regions affects the mechanical properties of the fibers.

Generally an increase in crystalline regions or degree of crystallinity leads to a tight molecular packing, an increase in fiber strength and brightness but a decrease in flexibility, moisture absorption and dyeability (2).

Cellulose has a mono-clinical crystal structure and size of the unit cell and angles between the edges of the unit cell are as follows (3):

a = 0.82 nm b = 1.03 nm
c = 0.78 nm $\alpha = \gamma = 90^{o}$ $\beta = 84^{o}$

For cellulose and regenerated cellulose structures hkl Miller indices are determined as 101 10$\bar{1}$ 002 (Figure 1) (4).

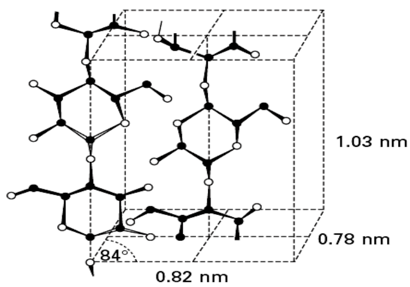


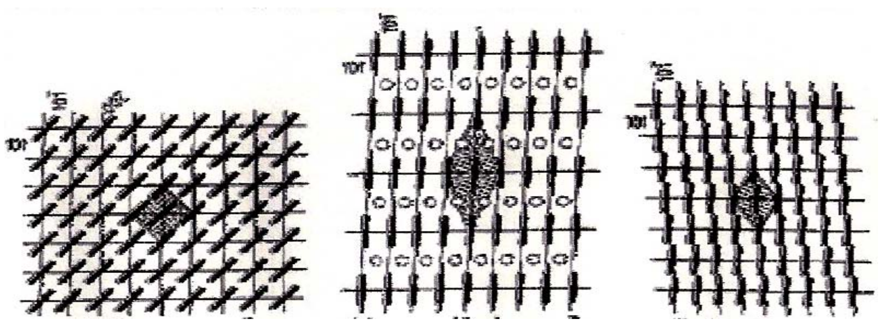
Figure 1. Crystal structure of cellulose (5)

Cellulose, the raw material of bamboo has different crystalline configurations. Natural products (cotton) have cellulose I, and regenerated cellulose products (bamboo, lyocell, viscose) have cellulose II structure. Cellulose I exists as I_{α} and I_{β} modifications depending on the source of cellulose. During regeneration process, NaOH application and swelling causes a conversion in the cage crystal structure from cellulose I to cellulose II. Each configuration has a different number of intramolecular and intermolecular H bridge bonds. Intramolecular hydrogen bridge bonds provide chain rigidity. As cellulose II has half as much hydrogen bridge bonds as cellulose I, cellulose II structured fibers have higher flexibility and lower strength than cellulose I structured fibers (Figure 2-3) (6, 7).

Bamboo, as a regenerated cellulosic fibre, is being more widely used in the textile industry due to its features such as being antibacterial, soft feeling, easy dyeability, absorbancy, breathability and having a smooth texture. Bamboo fiber has wide prospects in the field of hygiene products, medical suppliers, such as wet wipe, household wipes, baby diper, sanitary napkin, medical bandage, disposable sheet, inside lining, base cloth, nonwoven textiles, nanotechnological products and so on (8-14). It's also the only 100% biodegradable textile material which does not cause any environmental pollution naturally recycling itself (8-14). In this sense, it is praised as the new environmentally friendly textile material.

There have been few researches carried out on the bamboo fibre; dyeing property of bamboo/wool blended fabric (15-16), physical and mechanical properties of bamboo fiber (17-20).

Table 1 lists the physical properties of bamboo and cotton fibers



Cellulose I **Na-Cellulose I** **Cellulose I**

Figure 2. Structıre of Cellulose I, Na-Cellullose I, - Cellulose II

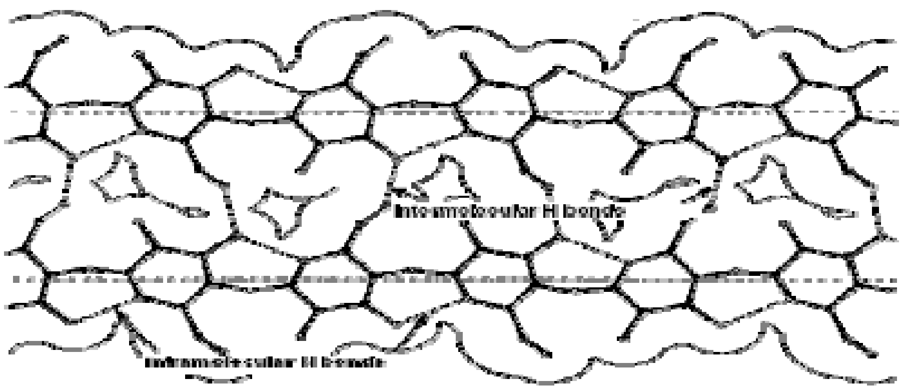


Figure 3. Intra and intermolecular H-bonds of cellulose

Table 1. Physical properties of the bamboo and cotton fiber (8, 21-24)

Properties	Bamboo	Cotton
Linear density (dtex)	1.67	1.8
Dry tensile strength (cN/tex)	22~25	24-28
Wet tensile strength (cN/tex)	13~17	25-30
% elongation – dry	14~18	7-9
Density (g/cm^3)	1.32	1.54
Moisture regain (%)	13	8.5

Regenerated bamboo fibers have lower tensile strength and higher elongation and moisture regain capacity compared to cotton fibers

In 21st century the importance of bamboo is increasing as a textile material. However, the researches on bamboo structure are limited. In this study, to have an in-depth knowledge of bamboo fiber the use of which is increasing in Turkey and many countries, an external structure analysis using SEM and an internal structure analysis by calculating percent crystallinity, crystal size, and degree of polymerization were carried out; the values obtained were compared with those of cotton fiber and the results were evaluated.

2. MATERIALS AND METHODS

100% regenerated bamboo and cotton knitted fabrics with similar properties were used in the studies. 100% knitted bamboo and cotton fabric were used throughout the experiment (300 stitch/cm^2 and ~130 g/m^2- Bilkont Dış Ticaret ve Tekstil Sanayi).

The bursting strengths of fabrics were determined by SDL ATLAS M 229 bursting test device conforming to the TS 393 EN ISO 13938-1 standard (25), and abrasion resistance test was carried out with NU-Martindale (Model 103) device conforming to the TS EN ISO 12947-2 /April 2001 standard (26).

The absorbency of fabrics were tested according to AATCC Test Method 79 (2007) (27).

The surfaces with a cross sectional study of fibres were observed by using a scanning electron microscope (SEM, **JEOL JSM-5910 LV**).

The fabrics were scanned and recorded using the D/MAX - 2200 X-ray diffractometer (XRD) (RIGAKU), using Cu-Kα radiotion generated at 40mA and 40 kV.

The crystallinity of cellulose samples were calculated from the X-ray diffraction patterns by the following equation

$$X_c\% = (I_{002} - I_{am}) x100 \ / I_{002} \qquad (1)$$

where I_{002} is the peak intensity from the (002) lattice plane and I_{am} the peak intensity of amorphous phases (28-29).

Apparent crystallite size (ACS) was calculated with Scherrer equation (2) by using the values of maximum intensity peaks in the XRD diffraction pattern (30-31).

$$ACS = 0.9\lambda / \beta \cos\theta \qquad (2)$$

where λ is the wavelength of the incident X-ray (1.5418 Å), θ the Bragg angle corresponding to the (002) plane, and β the half-height width of the peak angle of the (002) reflection.

Degree of polymerization (DP) of the cotton and bamboo fiber were ***calculated*** from the intrinsic ***viscosity*** using the following Mark-Houwink equation (32-34)

$$(\eta) = k_p \, DP_v^{\alpha} . \qquad (3)$$

The equation gives a relation between intrinsic viscosity (η) and DP. The values α and k_p depend on the polymer-solvent (cellulose – cuen) system (35-38).

3. RESULTS AND DISCUSSION

In this study, the crystal structure of the fibers was investigated by SEM and XRD.

SEM images

Figure 4 and 5 demonstrate the cross-sectional and longitudinal images of bamboo fibers.

It can be seen that the surface of the bamboo fiber has a non-circular lobed structure and there are many grooves and holes on it.

The porous structure of bamboo fibers are presumed to be responsible for their high water absorption capacity. The micropores and spaces in the cross-section of the bamboo fiber provide breathability and cool feeling to the bamboo fiber.

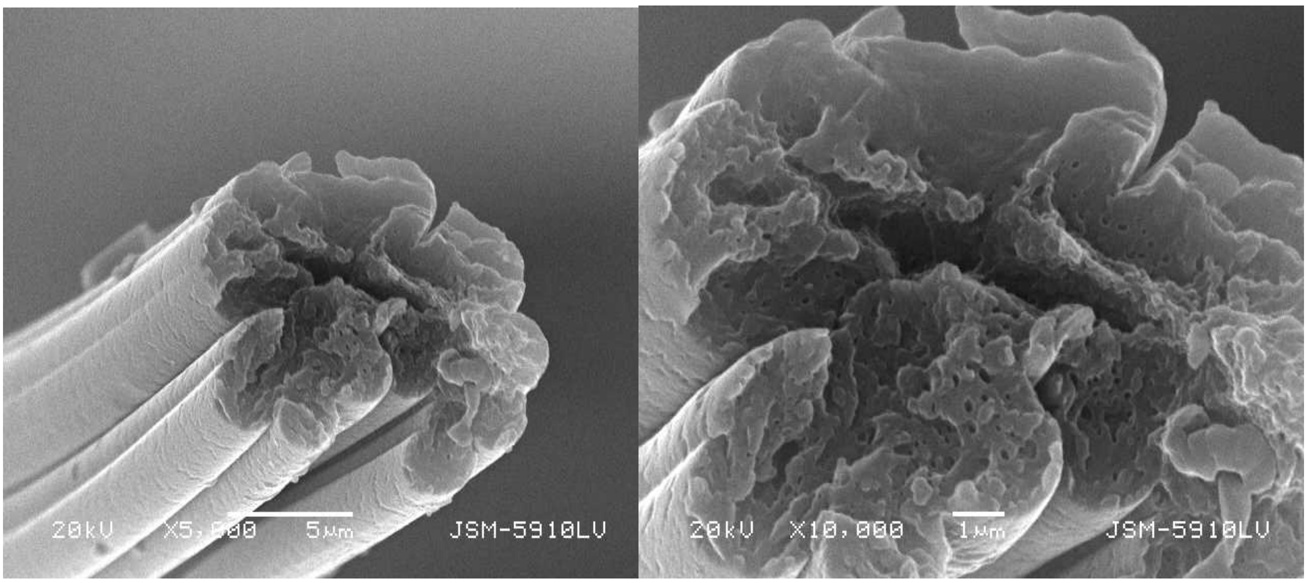


Figure 4. The cross-sectional images of bamboo fibers (Magnification 5,000x and 10,000x)

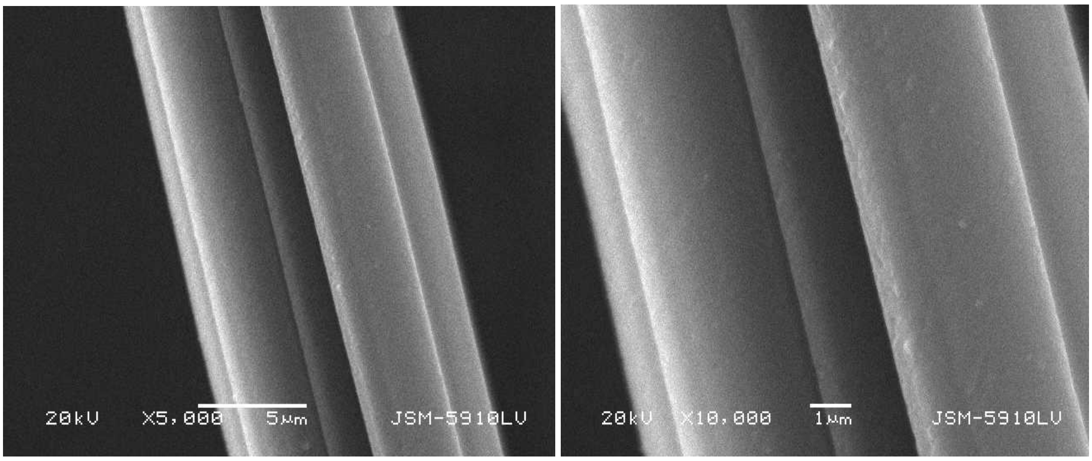


Figure 5. The longitudinal images of bamboo fibers (Magnification 5,000x and 10,000x)

Degree of Polymerization

The internal structure of the bamboo fiber was examined; crystal size, % crystallinity value using the drawn x-ray diffraction pattern (Figure 6) and degree of polymerization of bamboo fiber was ***calculated.*** Moreover, the mechanical properties (***bursting*** strength, abrasion ***resistance*** and absorbency) of the bamboo fiber were studied (39). The same tests and calculations were performed for the cotton fiber and the results were compared (Table 2).

When the results were evaluated, the DP of bamboo was found to be approximately one fifth of that of cotton.

Table 2. DP of bamboo and cotton fibre

	(η) (dL/g)	DP
Bamboo	3.674	490
Cotton	16.109	2147

Crystallite size (ACS)

Using XRD device, diffraction patterns of 100% bamboo and cotton knitted fabric were obtained and crystal size and percent crystallinity values were calculated and evaluated.

As the β value in the Scherrer equation (2) is the full width at half maximum (FWHM) of the peak obtained in the diffraction pattern (in radians), with increasing particle size, the peaks indicating the reflected beams in the diffraction pattern get narrower.

As the radiation source for XRD analysis was Cu K_α, a λ value of 1.5402 Å was used in calculations and the required values are shown in Table 3.

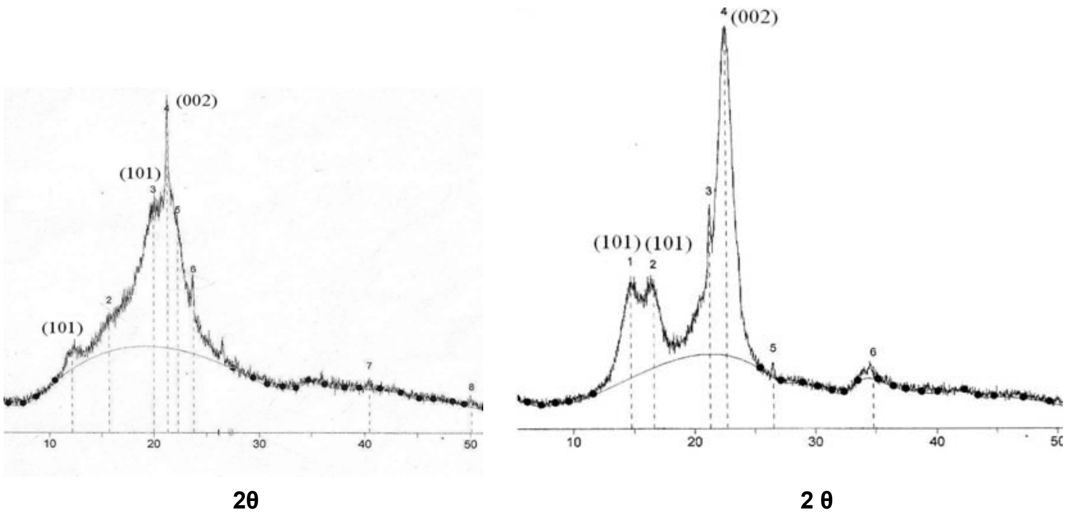


Figure 6. X-ray diffraction pattern a) 100% bamboo fiber b) % 100 cotton fiber

Tablo 3. Crystallite size of bamboo and cotton fiber

	hkl	2θ	FWHM	Crystallite size(Å)
Bamboo	***002***	21.338	1.059	**77**
Cotton	***002***	22.700	1.176	**69**

When particle sizes at 002 peak values were evaluated, the crystallite size of bamboo fiber was found to be larger than that of cotton fiber with a ratio of 1.1.

% Crystallinity

The percent crystallinity for raw bamboo and cotton were calculated by two different methods with intensity versus 2θ plots and the values obtained were compared.

1. method

In this method, calculations were performed using the integral areas at Figure 7-9 and equation (4).

X_C% = (crystalline area / the whole area) x 100 (28) (4)

%Crystallinity of the bamboo fibers (%X_{CB1})

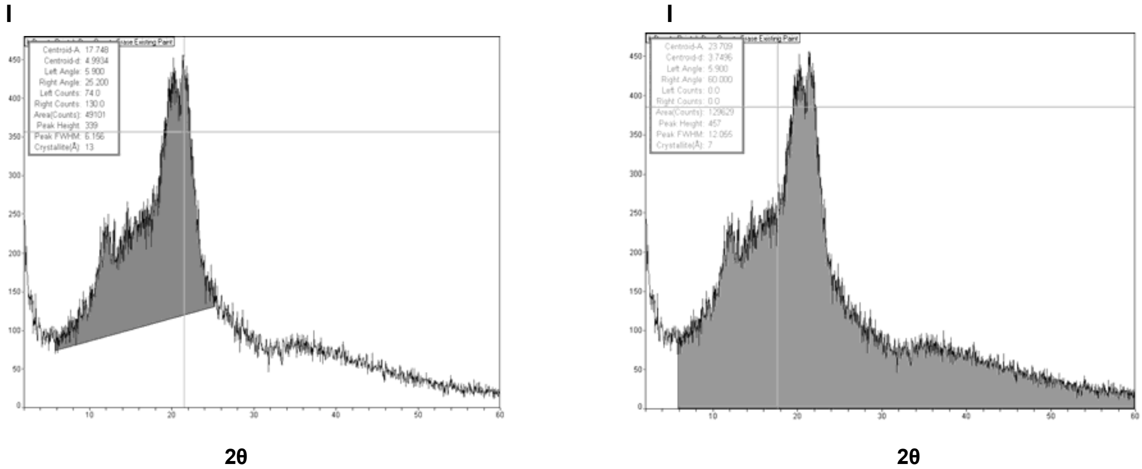


Figure 7. a- Crystalline area of bamboo b- The whole area of bamboo

Crystalline area (5.9 º - 25.2º) = 49101 The whole area (5.9 º - 60.0º) = 129629 %X_{CB1} = (49101 / 129629) x 100 = 37.87

%Crystallinity of the cotton fibers (%X_{CC1})

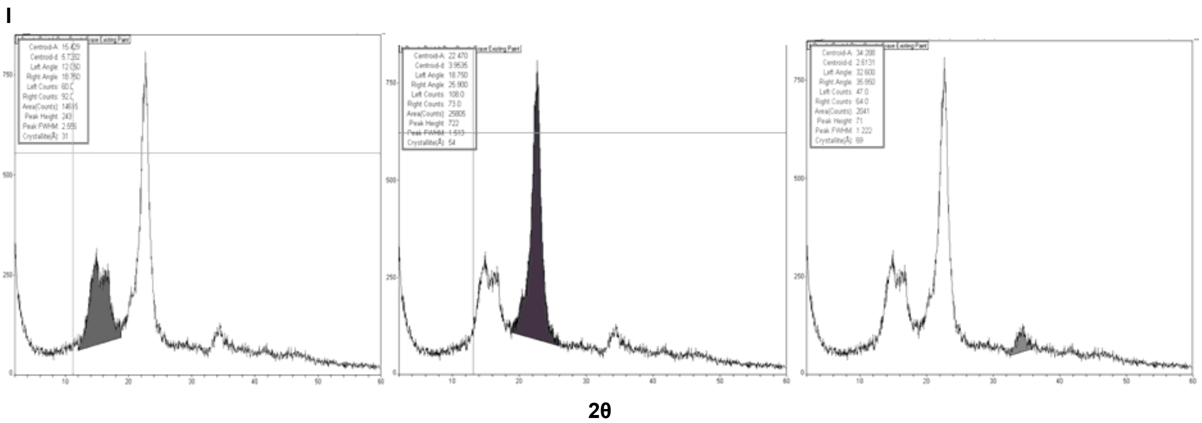


Figure 8. Crystalline areas of cotton (X_C)

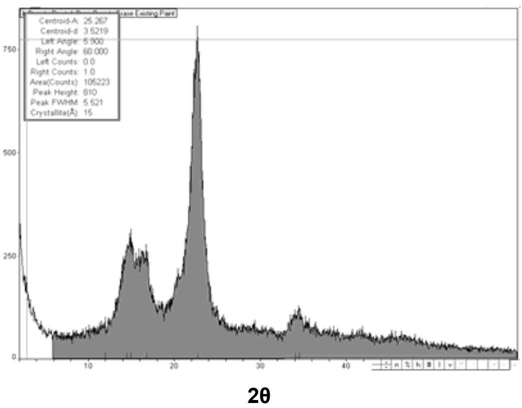


Figure 9. The whole area of cotton (X_{Cw})

X_{C1} (12.05 º - 18.75 º) = 14615 X_{C2} (18.75º - 25.90 º) = 27174 X_{C3} (32.61º - 35.85 º) = 2041

The whole area = (5.9 º - 60.0º) 105223 %X_{CP1} = (43830/ 105223) x 100 = 41.65

Table 4. % Crystalline values of bamboo and cotton fiber

Material	Hkl	2θ	BG	Heigh(I)	Heigh(%)	$\%X_c$
Bamboo	002	21.338	1187	690	100.0	36.76
Cotton	002	22.700	1065	1281	100.0	54.60

2. method

However in this method, percent crystallinity was calculated using the maximum peak heights obtained from X-ray diffraction patterns of bamboo and cotton knitted fabric samples and equation (5) (40-41).

$\%X_C=(I_{crystalline}/I_{amorphous}+I_{crystalline})x100$ (5)

The results of percent crystallinity calculations of bamboo and cotton fiber samples performed using two different methods are shown in Table 4. The results indicate that percent crystallinity values of bamboo fiber - which is a regenerated cellulose- are lower than that of cotton fiber, and the ratio of percent crystallinity of cotton fiber to bamboo fiber varies from 1.1 to 1.4.

The overall results of tests and calculations performed on bamboo and cotton knitted fabric samples are presented in Table 5.

Table 5. % Crystallinity of bamboo and cotton fiber

	% Crystallinity	
	Bamboo	**Cotton**
1. method	37.87	41.65
2. method	36.76	54.60

The subjective BG values used in X-ray diffraction patterns obtained for the samples lead to discrepancies in the calculated percent crystallinity values. In order to eliminate this subjectivity, we considered increasing the number of tests or calculating the percent crystallinity for another sample under standard conditions and taking the ratio of the percent crstallinity of the two samples.

Table 6. Crystal structure and mechanical properties of the bamboo and cotton fiber.

	Bamboo	Cotton
DP	724	3742
Crystal size (Å)	77	69
% Crystallinity	36.76	54.60
Bursting strength (kPa)	351.0	569.9
Abrasion resistance (Tour)	102000	28000
Absorbency (s)	<1	60<

The analyses performed demonstrated that the polymerization degree and % crystallinity value of bamboo fiber were lower than that of cotton and crystal size of bamboo fiber was higher than that of cotton. These results can effect the bursting strength of the bamboo fabric negatively (4). This suggests that the bursting strength of bamboo fabric is much lower when compared to cotton fabric. On the other hand, it was concluded that the non-circular lobed structure of the bamboo fiber reduces the friction area and affects the abrasion resistance of the fabric positively (39).

The larger crystal size of bamboo fiber compared to cotton fiber is thought to arise from the abundance of amorphous regions in bamboo fiber. As demonstrated in Table 6, the crystal size of bamboo fiber is 10% larger than that of cotton fiber and the percent crystallinity of cotton fiber is 10% higher than that of bamboo fiber.

In conclusion, internal structural differences may explain the differences in the mechanical properties of cellulose bamboo and cotton fibers.

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