

Contributions to the Study of the Lyocell Fiber Properties

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Lyocell fibers are ecological cellulose regenerated fibers and represent the most important discovery in the field of "artificial fiber engineering". These fibers can be situated at the border between natural fibers (in terms of comfort, biodegradability) and chemical fibers (by high resistance in dry and wet environment, low contraction). In order to characterize the structural Lyocell fiber properties, electronic microscopy analyses, birefringence and X-ray diffraction have been accomplished. The present analysis on the structural properties of Lyocell fibers allowed to conclude that the microscopic images depict that Lyocell fibers present uniform longitudinal aspect and regular cross-section; the Lyocell fiber birefringence is 1.6 times higher than similar feature of viscose fiber (0.04263 as compared to 0.0265) and the diffraction analysis reveals that the distortion degree of the crystal lattice of Lyocell and viscose fibers is in accordance with the crystalline factor. The average tenacity – deformation diagrams for Lyocell and viscose fibres, in conditioned and in wet medium, allowed to determine the rheological parameters of the fibers.

Keywords: Lyocell fiber properties, electronic microscopy, birefringence, diffraction analyses

The development of Lyocell fibers was driven by the desire for an environmental friendly process in order to produce cellulosic fibers with low cost and improved performances in comparison with viscose rayons [1]. The first commercial Lyocell fibers were available in 1984. The manufacture process consists of the direct dissolution of wood pulp in N-methylmorpholine-N-oxide (NMMO) and water at elevated temperatures [2]. The novelty of the process was the dissolving of cellulose in an organic solvent without derivation. This difference being found in the NMMO process and other processes has led to the new generic name of "Lyocell". It represents the newest and the most important discovery in the field of "artificial fiber engineering". Lyocell fibers have a unique property profile; they can be situated at the border between natural fibers (in terms of comfort, biodegradability) and chemical fibers (in terms of high resistance in dry and wet environment, low contraction). The Lyocell fabrics and garments have an excellent stability in wet state, good dyeability, brilliant shades, and soft drape. The garments are proper for wearing them directly on the skin, having thermophysiological and skin sensory properties. The Lyocell fibers present the tendency of fibrillation in a wet environment, opening the possibility of modifying the textile surface (e.g. the peach skin effect) [3-5]

In literature one can find some information about the Lyocell fiber properties, especially the structural

description. This is the reason for which this paper is focused on analyzing and characterizing the Lyocell fiber properties in the structural point of view. In this respect, electronic microscopy, birefringence and X-ray diffraction analysis have been accomplished.

The structural properties of Lyocell fibers were studied and analyzed in comparison with viscose fibers.

Experimental part

The Lyocell fiber samples used in this research were supplied by Lenzing AG Company.

The morphological characterization of Lyocell fiber has been done by electronic microscopy analysis. The microscopic images of the longitudinal aspect and cross section of the fibers were performed with a STEREOSCAN 250 apparatus.

In order to complete the picture on Lyocell fiber properties in comparison with the viscose fibers, birefringence analysis was performed and the orientation factors were established. In order to measure the birefringence of the two fibers, the minimum, medium and maximum diameters have been determined and the average diameters have been also evaluated (table 1).

Depending on diameters, birefringence measures have been accomplished (Dn), on an Amplival apparatus.

For determining the structural parameters of Lyocell fibers, X-ray diffraction analysis was performed.

Table 1
RESULTS OF DIAMETER MEASURES FOR THE LYOCELL AND VISCOSE FIBERS

Type of fiber	d_{min} (μm)	d_{med} (μm)	d_{max} (μm)	\bar{d} (weighed average) (μm)
	calculated from the distribution after frequency curve			from 250 values
Lyocell	8	12	16	12,12
Viscose	8	12	16	11,83

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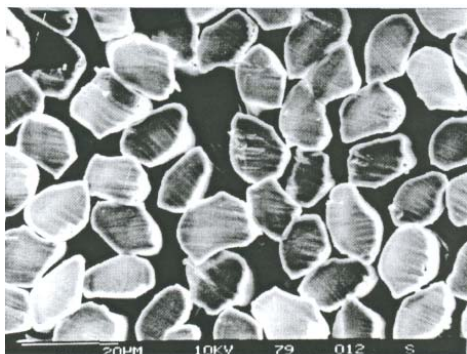


Fig. 1. Cross section of the Lenzing Lyocell fibers

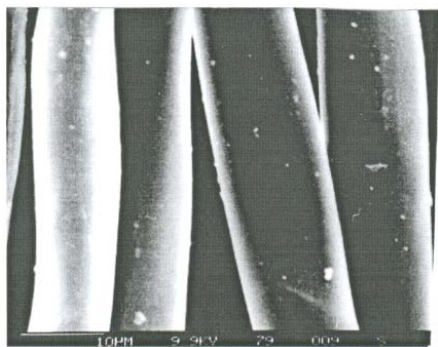


Fig. 2. Longitudinal aspect of the Lenzing Lyocell fibers

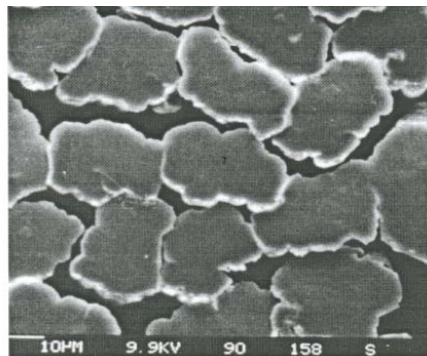


Fig. 3. Cross-section of the viscose fibers

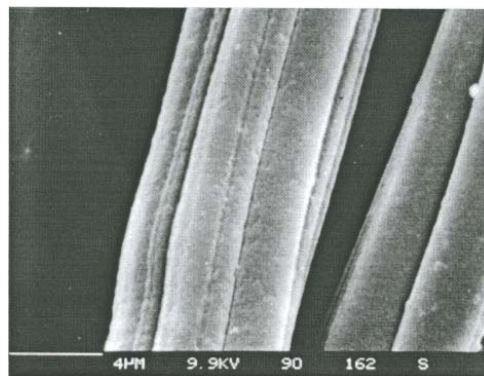


Fig. 4. Longitudinal aspect of the viscose fibers

The viscose and Lyocell samples were investigated by X ray diffraction using a $\text{CuK}\alpha$ filtered radiation and a pass of $0.01\ 2\theta$ [6]. The $\text{CuK}\alpha_1$ and $\text{CuK}\alpha_2$ components were separated by known methods [7, 8], the positions of the diffraction maximums and of the semiwidth of diffraction maximums for $\text{CuK}\alpha_1$ and $\text{CuK}\alpha_2$ components being determined with an accuracy from 0.02 up to $0.15\ 2\theta$, function of the semiwidth of the diffraction maximum and the ratio signal/noise. Corrections of the experimental data and indices of diffraction maximums were applied according to the Herman's and Weidinger's results [9]. The semiwidth diffraction maximums were corrected for the instrumental effect and finally, the physical component of the maxima was obtained. The correction was achieved supposing a Gauss type shape of the diffraction line, a fact confirmed by filtering diffraction maximums based on some type of functions (Gauss, Lorentz, Cauchy etc):

$$\beta = \sqrt{B^2 - b^2} \quad (1)$$

where:

B – is the corrected semiwidth for the $\text{CuK}\alpha_1$ and $\text{CuK}\alpha_2$ and corresponds to the pattern maximum and

b – is the semiwidth component of the diffraction line and is caused by the geometry of measuring system.

From the obtained data one can see that the most important contribution to the semiwidth line is that of the micro-distortions of the crystal lattice (ε), which was evaluated by applying the equation (2):

$$\varepsilon = \frac{\beta}{4tg\theta} \quad (2)$$

where θ represents Bragg angle corresponding to the centroid of the diffraction maximum. The crystalline factor was defined as the ratio between the total area of the crystalline maximums and the total area of the corrected diffraction patterns according to the Herman's and Weidinger's procedure [9, 10].

Taking into account the determination of the main evaluating parameters of fibre quality, the component elements of the strain/elongation diagrams and diagram tracing, there were performed standard mechanical tests for conditioned Lyocell and viscose fibres of $1.7\ \text{dtex}/38\ \text{mm}$, in a wet medium and loop. This analysis was carried out on a Textechno Fafegraph ME/Germany apparatus.

According to the provisions in the above mentioned standard tests, there were performed 100 determinations for the analysis of the conditioned fibres, and 50 determinations for analysis of fibres in wet state and loop [14].

Results and discussion

In figures 1-4, the cross section images and, respectively, the longitudinal aspects of Lenzing Lyocell and viscose fibers are represented.

The shape of cross sections is different for Lyocell fibers and the viscose ones. While the shape of Lyocell fibers is regular, the viscose fibers have a crenellated cross section profile [11, 12].

In accordance with the presented images, one can see that the Lyocell fibers present:

- a uniform longitudinal aspect. This characteristic is responsible for the good mechanical and tinctorial properties of the Lyocell fibers [7];

- a regular cross-section. The regular shape of the cross section in the case of Lenzing Lyocell fibers has an important contribution to their high tenacity in a dry/wet environment, low elongation and good dimensional stability [11 - 13, 15, 16].

The results of the birefringence measures are presented in table 2.

The obtained results allowed the following remarks:

- the birefringence of Lyocell fibers is a variable function of the fiber thickness: the thinner fibers are more oriented than those with medium or maximum diameter; this fact

Table 2
THE BIREFRINGENCE VALUES FOR THE LYOCELL AND VISCOSE FIBERS

Type of fiber	Δn (for d_{\min})	Δn (for d_{med})	Δn (for d_{\max})	$\Delta \bar{n}$ *
Lyocell	0,04201	0,04136	0,04112	0,04263
Viscose	insignificant differences as a function of variable thickness			0,0265

* average of 7 determinations

can be explained by the crimping degree in the cable. With viscose fibers, the differences are insignificant [4];

- the Lyocell fiber birefringence is 1.6 times higher than that of the viscose fibers (0.04263 as compared with 0.0265), which represents a high degree for a fiber, having in view the fact that the theoretical birefringence of a perfect crystal is 0.047 [5, 6].

For obtaining the orientation factor, the formula is:

$$f = \frac{\Delta n_d \cdot \rho_o}{\Delta n_o \cdot \rho_d} \quad (3)$$

where:

- Δn_d = calculated birefringence;
- ρ_d = determined density (g/cm^3);
- Δn_o = theoretical birefringence of a perfect crystal (0.047);
- ρ_o = theoretical density of a perfect crystal 1.58 g cm^3 , crystalline density for cellulose II.

This means that the orientation factor for the Lyocell fibers is:

$$f = \frac{0,04263}{0,047} \cdot \frac{1,58}{1,557} = 0.9204 \quad (4)$$

The orientation factor for viscose is expressed by the equation (5):

$$f = \frac{0,0265}{0,047} \cdot \frac{1,58}{1,533} = 0.5811 \quad (5)$$

One can mention the fact that the density has been determined by the geometrical method.

The obtained results allowed the following remarks:

- the orientation factor of Lyocell fibers is higher than that of viscose fibers (0.9204 for the Lyocell fibers and 0.5811 for viscose fibers), the fact that explains the high resistance of these fibers as compared to viscose fibers;
- the Lyocell fibers have a higher density as compared to the viscose ones (1.557 for the Lyocell fibers and 1.533 for the viscose fibers), as a result of a higher orientation degree.

The diffraction patterns of Lyocell and viscose are represented in figure 5 and the data regarding the corrected interplanetary distances, the Miller indices, and the distortion degree of the crystal grating are presented in tables 3 and 4. It results that the distortion degree of the crystal lattice of Lyocell and viscose fibers is in accordance with the crystalline factor. The increasing of the crystal degree can be associated with a decreasing of the distortion degree of the samples. An additional maximum is present in the Lyocell pattern suggesting a blend of more cellulose types in the pattern.

By the average tenacity – deformation diagrams for the Lyocell and viscose fibres in conditioned and wet medium (fig. 6 – 9), there were determined the initial modulus, the flow modulus and the coordinates of the proportionality, elasticity, flow and break limits, which are listed in table 5.

The determination of the parameters of the tenacity – deformation curves allow to trace the average tenacity – deformation diagrams for Lyocell fibres and respectively viscose, in conditioned state and wet medium.

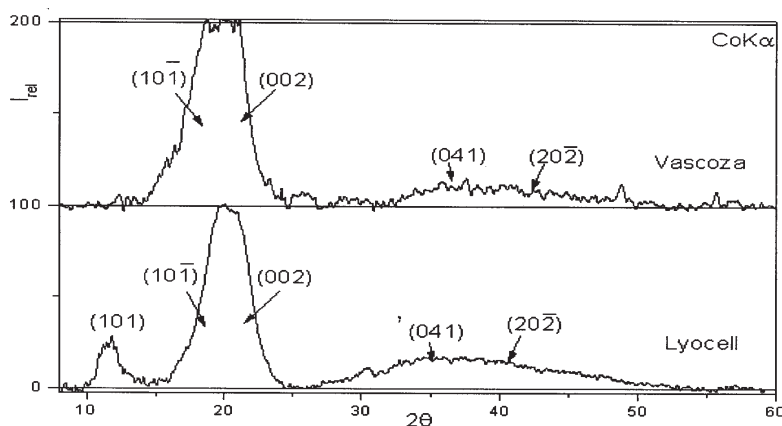


Fig. 5. The diffraction patterns of viscose and Lyocell

Table 3
CRYSTALLINE PARAMETERS OF VISCOSE

$2\theta_{\text{obs}}$	hkl	$d^{1)}$	$\epsilon^{3)}$	$f_c^{2)}$
11.68	101	8.462		0.96
20.05	10^{-1}	5.026	0.073	
21.43	002	4.708		
34.22	040	3.003	0.106	
40.36	20^{-2}	2.566		

¹⁾ corrected interplanetary distances;

²⁾ crystalline factor;

³⁾ micro-distortions of the crystal grating.

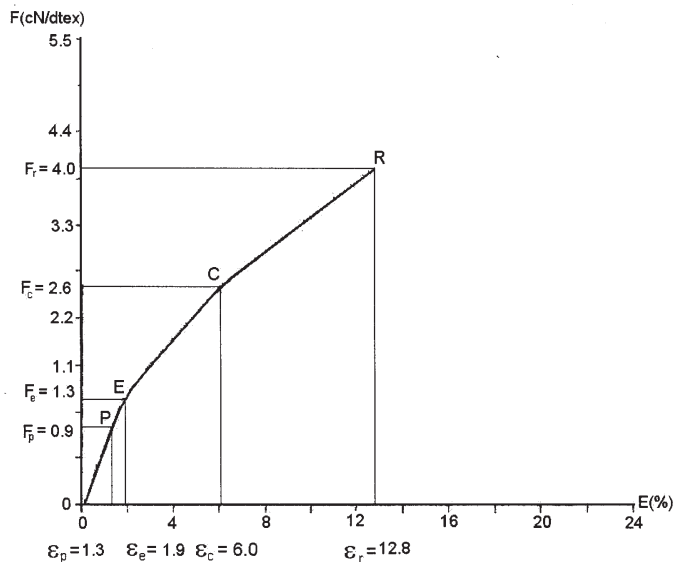


Fig. 6. Average tenacity – deformation diagram for the Lyocell fibres – in conditioned medium

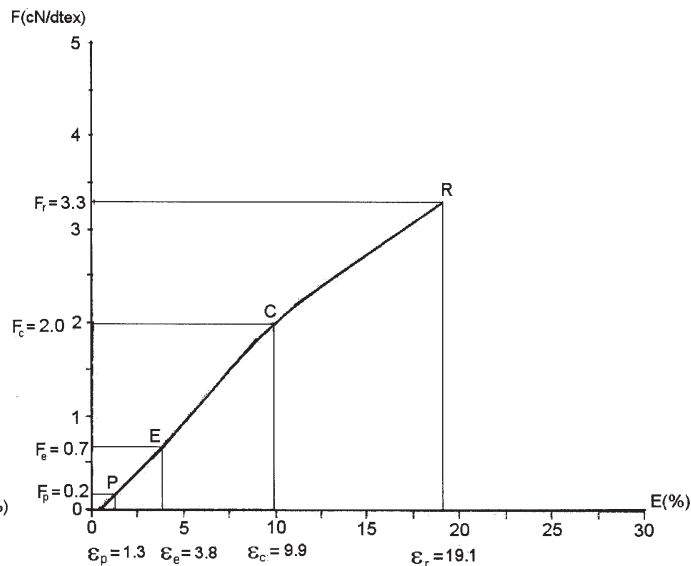


Fig. 8. Average tenacity – deformation diagram for the Lyocell fibres – in wet medium

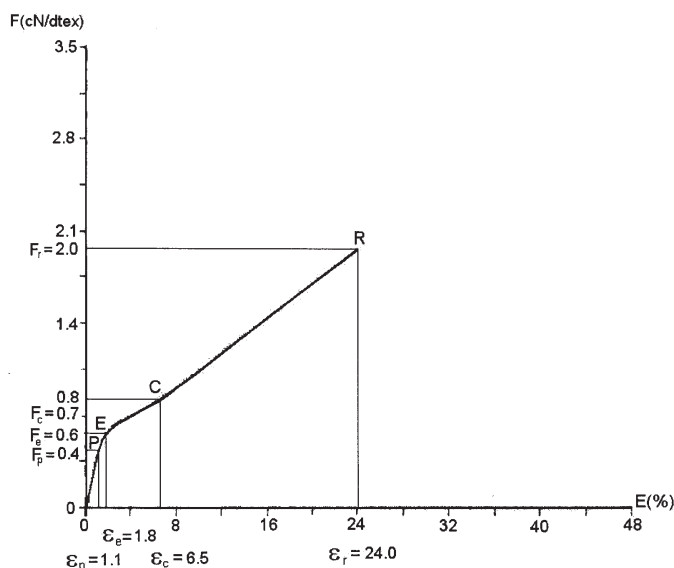


Fig. 7. Average tenacity – deformation diagram for the viscose fibres – in conditioned medium

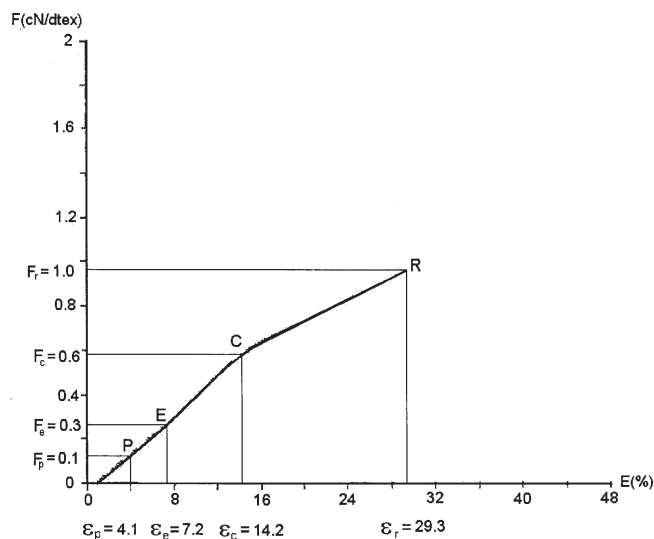


Fig. 9. Average tenacity – deformation diagram for the viscose fibres – in wet medium

The analysis of the tenacity-deformation curves and the parameters that are peculiar to these diagrams in the case of Lyocell and viscose fibres, in a conditioned medium, have allowed the highlighting of the following aspects:

- the average strain – elongation curves have different shapes, owing to the various break resistances, which come from the structural differences between the two fibre types;

- the domain of elastic deformations is more than two times larger for the Lyocell fibres, as compared to that of the viscose ones. With Lyocell fibres, the elasticity limit has the E coordinates (1.3;1.9), as compared to E (0.6;1.8) for the viscose ones, a fact that can be explained by the higher degree in crystallinity and molecular orientation;

- between the proportionality limit and flow limit (the flow zone or creep), delimited by the flow limit C, one can see a high deformation capacity in both fibre types, when under relatively low strain: $C_{Lyocell}$ (2.6;6) and $C_{viscose}$ (0.8;6.5) are obtained;

- the break limit R marks the macroscopic destruction of the fibres, with a tenacity of 4 cN/dtex (40cN/tex) for the Lyocell fibres, in comparison with a tenacity of 2 cN/dtex (20 cN/tex) for the viscose fibres;

- the elongation is quite similar for both fibre types up to the elasticity limit; important longitudinal deformations take place starting with the flow zone up to the break limit, where the fibres reach an elongation that is about two times larger for Lyocell fibres in comparison with viscose fibres (24% as compared to 12.8%, which represents an increase by 53.3% for Lyocell fibres).

By means of the analysis of Lyocell and viscose fibre behaviour under strain, in a wet medium, with the help of the strain – elongation diagrams, the following aspects are to be noticed:

- the initial portion of the tenacity – elongation curves linearly increases up to point E, which marks the elastic limit, then there appears a slight decrease of the slope, which can be assimilated to the flow; after that the break takes place, without any slope modification (without any increase, as in the tests performed in a conditioned medium). It results that the analysed Lyocell and viscose fibres have only the elasticity and break limit (the flow limit no longer appears, the fibre break taking place directly), a fact which was also brought forward in table 5;

- the average strain – elongation curves have different shapes, because the crystallinity and fibre orientation

Table 4
CRYSTALLINE PARAMETERS OF LYOCELL FIBER

$2\theta_{obs}$	hkl	$d^{1)}$	ε	$f_c^{2)}$
19.27	10^{-1}	5.224	0.095	0.92
21.01	002	4.805		
35.50	041	2.899	0.094	
42.68	20^{-2}			

Table 5
DETERMINED PARAMETERS OF THE TENACITY – DEFORMATION CURVES

Fibre	Initial modulus	Flow modulus	Proportionality limit		Elastic limit		Flow limit		Break limit	
	E_i cN/tex	E cN/tex _c	σ_p cN/tex	ε_p %	σ_e cN/tex	ε_e %	σ_c cN/tex	ε_c %	σ_r cN/tex	ε_r %
Conditioned Lyocell	690	317	9	1.3	13	1.9	26	6	40	12.8
Conditioned Lyocell	184.2	141.3	-	-	20	9.9	-	-	33	19.1
Conditioned Lyocell	363.6	42.55	4	1.1	6	1.8	8	6.5	20	24
Conditioned Lyocell	41.6	26.5	-	-	6	14.2	-	-	10	29.3

degree influences the break and break elongation resistances, so that Lyocell fibres that have a higher crystallinity degree will swell in water to a lower degree and, therefore, will have a slighter resistance decreasing in comparison with the viscose ones;

-the domain of the elastic deformations is more than three times higher for the Lyocell fibres, as compared to the viscose ones; with Lyocell fibres, the elasticity limit has the coordinates E (2.0; 9.9), as compared to E (0.6; 14.2) with the viscose ones, a fact that justifies itself by the different morphologic structures of the two fibre types;

-the microscopic fibre destruction appears at a 3.3 times higher tenacity for Lyocell fibres, by comparison with the viscose ones (3.3 cN/tex as compared to 1.0 cN/tex).

Conclusions

In order to characterize Lyocell fibers structure, electronic microscopy, birefringence and X-ray diffraction analysis have been accomplished.

The microscopic images reflect the fact that Lyocell fibers present:

- a uniform longitudinal aspect, which means good mechanical and tinctorial properties of Lyocell fibers;

- a regular cross-section, which explains the high tenacity in dry/wet environment, low elongation and good dimensional stability of Lyocell fibers.

The obtained data after measuring the birefringence and the crystalline factor have emphasized:

- Lyocell fiber birefringence is 1.6 times higher than that of the viscose fibers (0.04263 as compared to 0.0265);

- orientation factor of Lyocell fibers is higher than that of the viscose fibers (0.9204 for Lyocell fibers and 0.5811 for viscose fibers), which explains the high resistance of these fibers as compared with the viscose fibers;

- Lyocell fibers have a higher density as compared to the viscose fibers (1.557 for Lyocell fibers and 1.533 for viscose fibers), as a result of a higher orientation degree;

The diffraction analysis reveals the fact that the distortion degree of the crystal lattices of Lyocell and viscose fibers are in accordance with the crystalline factor. The increasing of the crystal degree can be associated with a decreasing of the distortion degree of the samples.

By means of the average tenacity – deformation diagrams for Lyocell and viscose fibres, in conditioned and wet medium, there have been determined the initial modulus, the flow modulus and the coordinates of the proportionality, elasticity, flow and break limits.

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