

Interferometric Determination of the Birefringence of Thermo-Tropic Polyester Fibers and Its Copolymers of Structure (PCPT-*co*-CPO)

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ABSTRACT: The aim of this study is to determine the optical anisotropy of three different samples of thermo-tropic polyester fibers of structure Poly(chloro-1,4-phenylene terephthalate-*co*-4,4'-oxybisbenzoate) and its copolymers (PCPT-*co*-CPO). The molar fraction of disruptor units ([CPO]) ranging from 0.40 to 0.60. The variable wavelength interferometry, VAWI technique, was used to determine the birefringence and spectral dispersion properties of such of these fibers. The Cauchy's dispersion formula and its related constants were determined using the spectral dispersion curves of the birefringence. A mathematical formula was derived for direct measurement of the birefrin-

gence profile of highly birefringent polymer fibers using VAWI technique. The effect of varying copolymer molar fractions of the three samples of thermo-tropic polyester fibers was investigated throughout the spectral dispersion curves, the Cauchy's formula constants and the birefringence profiles. Microinterferograms are given for illustration. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 1814–1821, 2012

Key words: thermo-tropic polyester fibers; (PCPT-*co*-CPO); variable wavelength interferometry; birefringence profile; spectral dispersion curves

INTRODUCTION

There is now a considerable scientific and technological interest in nematogenic polymer.¹ Polymers, which can form a thermally stable nematic mesophase are called thermotropic nematics. They have received considerable attention because of their potential applications as high performance fibers. The most notable properties of thermotropic polyesters are their high modulus, low melt viscosity, high chemical resistance and low mould shrinkage.² The absence of shrinkage in the mould is a consequence of low coefficients of thermal expansion of these polymers. The coefficient parallel to the direction of orientation in oriented samples can even be negative behavior which can perhaps be attributed to higher rotational energy levels in the trans ester groups leading to a shorter average ester unit length in the direction of measurement.¹ Also, these properties tend to be anisotropic due to the fact that rigid chain thermotropic polyester unlike conventional polymers with chain folded lamellar crystals, tend to remain in an aligned structure even upon melting.²

Researchers interested in thermotropic polyesters concerned to the relation between the chemical structure and transition temperatures, but now there is a growing emphasis on characterization of physical structure.^{3–8}

One of the most characteristic features of the fiber is optical anisotropy, birefringence, which is necessary for the fiber structural properties on the molecular level. These properties are changed largely by the complicated factors acting during processing, starting with spinning and ending in the manufactures. The study of the birefringence in thermotropic polyester fibers plays an important role in the knowledge of the molecular arrangement within these fibers. It gives a measure of the orientation, which is the average of the amorphous and crystalline region.⁹

Interferometric techniques have long been invaluable techniques for studying the optical properties of polymeric birefringent fibers.^{10,11} The variable wavelength interferometry, VAWI,¹¹ is commonly used for measuring the spectral dispersion properties of the refractive indices and birefringence of the fiber material. Pluta,¹² Hamza et al.,^{13,14} and Sokkar et al.¹⁵ reported different methods to use VAWI technique for studying the spectral dispersion properties of different types of fibers. Sokkar and El-Bakary¹⁶ suggested a method for measuring the refractive index profile of homogeneous highly oriented

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fibers using the VAWI technique. El-Bakary^{17,18} used double refracting interference microscopy for determining the refractive index profile of highly oriented fibers¹⁷ and the VAWI technique to determine the radial structural properties and spectral dispersion curves of poly(aryl ether ether ketone) fiber.¹⁸

In this work, the VAWI technique was used to determine the optical anisotropy and spectral dispersion properties of Poly(chloro-1,4-phenylene terephthalate) fibers and its copolymers. The Cauchy's dispersion formula and its related constants were determined using the spectral dispersion curves of the birefringence. A mathematical formula was derived for direct measurement the birefringence profile of polymer fibers. The effect of varying copolymer molar fractions of the three samples of thermo-tropic polyester fibers of structure (PCPT-co-CPO) was investigated throughout the spectral dispersion curves, the Cauchy's constants and birefringence profiles.

MATERIALS

Three different samples of thermo-tropic polyester and its copolymer fibers of structure Poly(chloro-1,4-phenylene terephthalate-co-4,4'-oxybisbenzoate), (PCPT-co-CPO), were prepared as spun in Leeds textile laboratory.⁵ These are copolymer polyester fibers with different molar fraction of disruptor units ([CPO]) ranging from 0.40 to 0.60. Sample 1 (BOB-40 As-Spun) has a molar fraction of disruptor units [CPO] = 0.40 and its code is (BS16 SP13). Sample 2 (BOB-50 As-Spun) has a molar fraction of disruptor units [CPO] = 0.50 and its code is (BS9 SP7 230/240). Sample 3 (BOB-60 As-Spun) has a molar fraction of disruptor units [CPO] = 0.60 with no heat treatment. These samples are manufactured in Textile Industries laboratory, University of Leeds, Leeds, UK.

THEORETICAL CONSIDERATIONS

The spectral dispersion of the birefringence using VAWI technique

The variable wavelength interferometry, VAWI technique, depends upon the use of the two beam interference microscope designed by Pluta.¹¹ The intensity distribution I of the interference pattern that was given by this microscope may approximately expressed as follows¹¹:

$$I = I_{\max} \sin^2(\varphi/2) = I_{\max} \sin^2(\pi\delta/\lambda) \quad (1)$$

where φ and δ are respectively the phase shift and the optical path difference between the light components (\parallel and \perp) due to the fiber birefringence where

$$(\delta = \varphi\lambda/2\pi).$$

The relation between these interference patterns on one hand and the birefringence Δn of the fiber under study and the wavelength $\lambda_2 < \lambda_1$ of monochromatic light used on the other hand makes it possible to determine the spectral dispersion of the birefringence $\Delta n(\lambda)$ or discrete birefringence for given light wavelengths. Starting from a long wavelength permits us to select a particular wavelength λ_1 for which the coincidence case of VAWI technique. This situation can be expressed as:

$$\delta_1 = d\Delta n_1 = m_1\lambda_1, \quad (2)$$

where m_1 is an integer number referred to as the initial interference order, d is the fiber thickness. Further decreasing the wavelength by transverse sliding the interference filter leads to other particular wavelength $\lambda_2 < \lambda_1$ for which the anti-coincidence case of VAWI technique. By transverse sliding the interference filter other situation at particular wavelengths can be obtained. All these situations can be described as¹¹:

$$\delta_s = d\Delta n_s = (m_1 + q_s)\lambda_s = m_s\lambda_s, \quad (3)$$

where $s = 2, 3, 4, \dots, q_s = 0.5, 1, 1.5, 2, \dots$. It self-evident that q_s expresses the increment of the current interference order. $q_1 = 0$ for $s = 1$ and $m_s = (m_1 + q_s)$.

Also, the equation of the initial interference order as in VAWI technique is given as:

$$m_1 = q_s \frac{b_s}{b_1 - b_s}, \quad (4)$$

where b_s is the interfringe spacing corresponding to λ_s and b_1 due to λ_1 . The subscript s denotes the coincidence and anticoincidence number. Using eqs. (3) and (4) the spectral dispersion curves of fiber material can be determined.

Cauchy's dispersion relation

When the incident light vector is parallel to the fiber axis, the variation of the refractive index of the fiber material with the wavelength can be written from the known Cauchy's dispersion relation¹⁹ as:

$$n^{\parallel}(\lambda) = A^{\parallel} + \frac{B^{\parallel}}{\lambda^2} \quad (5)$$

where $n^{\parallel}(\lambda)$ is the refractive index at a given wavelength λ , A^{\parallel} and B^{\parallel} are constants depend on the fiber material and called Cauchy's constants. An analogue formula for the refractive index $n^{\perp}(\lambda)$

when the incident light vector is perpendicular to the fiber axis. The birefringence of the fiber material is the difference between the refractive index when light vector is parallel and perpendicular to the fiber axis ($\Delta n(\lambda) = n^{||}(\lambda) - n^{\perp}(\lambda)$). Using this definition, the following equation can be written as:

$$\Delta n(\lambda) = \Delta A + \frac{\Delta B}{\lambda^2} \quad (6)$$

where $\Delta n(\lambda)$ is the birefringence at a given wavelength λ , ΔA , and ΔB are constants of the Cauchy's formula in this case.

The birefringence profile

Variable wavelength interferometry, VAWI technique, was used for measuring the refractive index profile of highly oriented fibers by Sokkar and El-Bakary.¹⁶ The highly birefringence fibers introduce a large optical path length difference with the surrounding medium. The method concerned on determining the positions of coincidence of the fringe shift inside the fiber with the surrounding medium fringes. The refractive index profile $n(r)$ of the fiber, taking the refraction into consideration, can be measured, using the previously derived following equation¹⁶;

$$n(r) = \frac{Z_x \lambda}{2ab[1 - (r^2/n^2a^2)]^{1/2}} + n_L[(1 - (r^2/a^2))^{1/2} \times (1 - (r^2/n^2a^2))^{1/2} + (1 - (r^2/a^2))^{1/2}] \quad (7)$$

where Z_x is the fringe shift displacement related to the value x along the fiber layer of radius r . λ is the wavelength of the monochromatic light used, a is radius of the fiber, n is the relative refractive index, n_L is the surrounding medium refractive index and b is the interfringe spacing. The above equation can be used when light vector is parallel to the fiber axis, to determine $n^{||}(r)$, and is perpendicular to the fiber axis, to determine $n^{\perp}(r)$. Also, the above equation is used when using the VAWI method in case of crossed position of the interferometer for obtaining the duplicated image of the fiber material. To determine the birefringence directly, the interferometer is used in its subtractive position for obtaining a non duplicated image of the fiber material. To calculate the birefringence profile from the above equation, we use the well known formula $\Delta n(r) = n^{||}(r) - n^{\perp}(r)$ and the birefringence profile formula can be easily derived as follows:

$$\Delta n(r) = \frac{\Delta Z_x \lambda}{2ab[1 - (r^2/n^2a^2)]^{1/2}} \quad (8)$$

where ΔZ_x is the fringe shift displacement related to the value x along the fiber layer of radius r in the

case of direct measurement of the birefringence using VAWI technique. Therefore, the birefringence profile can be obtained, taking the refraction of the incident light beam by the fiber layer from eq. (8). The advantage of the method is determining the birefringence profile from the nonduplicated image directly without the calculation of the refractive indices $n^{||}$ and n^{\perp} .

EXPERIMENTAL TECHNIQUE

The automatic VAWI micro-interferometer²⁰ was used. This technique is specially designed to measure and study the spectral dispersion of the refractive indices and/or birefringence of fibrous material using VAWI method. The main part of the automatic optical system is the Biolar PI micro-interferometer. This microscope has two positions; crossed position for obtaining the duplicated images of the fiber under study and the subtractive position for obtaining the nonduplicated image of the fiber under test. The automatic system is fitted with a highly monochromatic light source, digital interference filter, stepper motor controller, PC computer with a CCD camera to transfer the image to the image analysis screen. The intensity of illumination of the interference field and the contrast of the fringes are optimized by adjusting the width of the condenser slit fitted in the microscope. The automatic measurements using this technique are controlled using the computer program menus and the calculations was done automatically using image analysis software program.

EXPERIMENTAL RESULTS AND DISCUSSIONS

Automatic measurement of the thickness and birefringence of thermo-tropic polyester fibers

The automatic micro-interferometer was adjusted in subtractive position²⁰ and the computer program menu was prepared for measuring the fiber thickness of three samples of thermo-tropic polyesters of structure (PCPT-co-CPO) with different molar fraction. The first sample was placed on the microscope stage and the program was used for automatic measurement of the fiber thickness. The average thickness of Sample 1 of copolymer [CPO] = 0.40 was found to be 27.36 μm , and for Sample 2 of copolymer [CPO] = 0.50 was found to be 24.32 μm . And for Sample 3 of copolymer [CPO] = 0.60 was found to be 32.68 μm .

For the measurement of the birefringence, the automatic micro-interferometer was adjusted in the subtractive position²⁰ and prepared, from the computer program menu, to obtain the non-duplicated image of the fiber under test. Three different

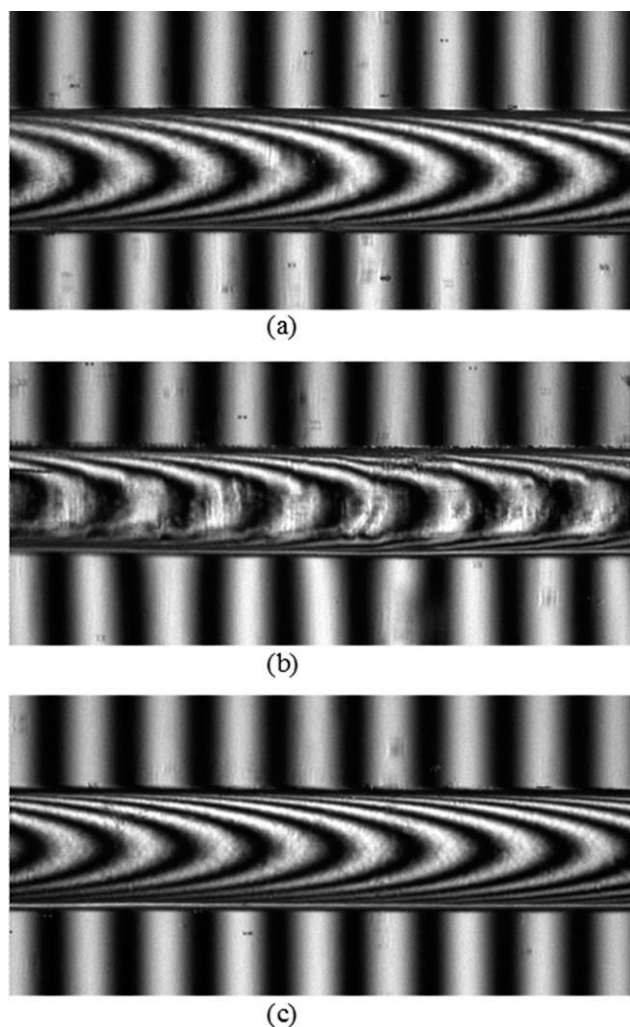


Figure 1 (a–c) The printed microinterferograms taken from the image screen of nonduplicated images of thermo-tropic polyester fibers (a) for the Sample 1 of copolymer [CPO] = 0.40, (b) for the Sample 2 of copolymer [CPO] = 0.50 and (c) for the Sample 3 of copolymer [CPO] = 0.60.

samples of thermo-tropic polyester of structure (PCPT-co-CPO) were examined at different wavelengths using the automatic VAWI technique. The

TABLE I
Results of the Measurements of the Birefringence of Sample 1 of Thermo-Tropic Polyester Fiber of Copolymer [CPO] = 0.40 and 27.36 μm Thickness Using VAWI Technique

s	q_s	b_s (μm)	$m(b)$	λ_s (nm)	m_s	δ_s (μm)	Δn
1	0	220.45	–	671.12	14	9.396	0.343
2	0.5	213.08	14.44	650.38	14.5	9.431	0.345
3	1	206.25	14.52	631.21	15	9.468	0.346
4	1.5	199.93	14.61	613.42	15.5	9.508	0.347
5	2	194.06	14.70	596.91	16	9.551	0.349
6	2.5	188.60	14.98	581.56	16.5	9.596	0.351
7	3	183.51	14.91	567.27	17	9.643	0.352
8	3.5	178.77	15.01	553.93	17.5	9.694	0.354
9	4	174.34	15.12	541.49	18	9.747	0.356
10	4.5	170.20	15.24	529.85	18.5	9.802	0.358

TABLE II
Results of the Measurements of the Birefringence of Sample 2 of Thermo-Tropic Polyester Fiber of Copolymer [CPO] = 0.50 and 24.32 μm Thickness Using VAWI Technique

s	q_s	b_s (μm)	$m(b)$	λ_s (nm)	m_s	δ_s (μm)	Δn
1	–0.5	223.65	12.59	680.10	12.5	8.501	0.349
2	0	214.77	–	655.15	13	8.517	0.350
3	0.5	206.91	13.16	633.05	13.5	8.546	0.351
4	1	199.96	13.49	613.50	14	8.589	0.353

first sample of copolymer [CPO] = 0.40 was placed on the microscope stage and the computer program was used to measure automatically the spectral dispersion curves of the birefringence of first sample. The measurement using this technique depending on defining the positions of coincidence and anti-coincidence of the fringes inside the fiber material with the surrounding medium fringes.¹¹ The same operation was done for Sample 2 of copolymer [CPO] = 0.50 and Sample 3 of copolymer [CPO] = 0.60. Figure 1(a–c) shows the printed microinterferograms taken from the image screen of nonduplicated images for the birefringence of thermo-tropic polyester fibers using automatic VAWI technique. Figure 1(a) for the Sample 1 of copolymer [CPO] = 0.40, Figure 1(b) for the Sample 2 of copolymer [CPO] = 0.50, and Figure 1(c) for the Sample 3 of copolymer [CPO] = 0.60.

The results of automatic measurements gives the number of coincidence and anticoincidence positions and different optical parameters as shown in Table I for Sample 1 of copolymer [CPO] = 0.40. Table II gives the results of the measurements for Sample 2 of copolymer [CPO] = 0.50. Also, the same optical properties were measured for Sample 3 of copolymer [CPO] = 0.60 and the results is shown in Table III. From these tables, it is clear that the measurements depend on determining the positions of coincidence and anticoincidence of the fringes inside the

TABLE III
Results of the Measurements of the Birefringence of Sample 3 of Thermo-Tropic Polyester Fiber of Copolymer [CPO] = 0.60 and 32.68 μm Thickness Using VAWI Technique

s	q_s	b_s (μm)	$m(b)$	λ_s (nm)	m_s	δ_s (μm)	Δn
1	–0.5	222.83	17.76	677.78	17.5	11.861	0.363
2	0	216.55	–	660.15	18	11.883	0.364
3	0.5	210.68	17.93	643.63	18.5	11.907	0.364
4	1	205.17	18.02	628.15	19	11.935	0.365
5	1.5	199.99	18.12	613.61	19.5	11.965	0.366
6	2	195.14	18.23	599.96	20	11.999	0.367
7	2.5	190.57	18.33	587.12	20.5	12.036	0.368
8	3	186.27	18.46	575.04	21	12.076	0.369
9	3.5	182.22	18.58	563.66	21.5	12.119	0.371
10	4	178.41	18.71	552.93	22	12.165	0.372
11	4.5	174.81	18.84	542.82	22.5	12.214	0.374

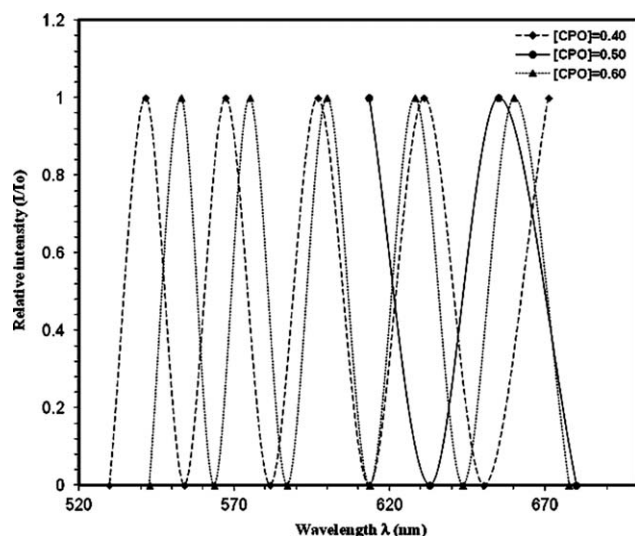


Figure 2 The relation between the relative intensity distribution of and the wavelength of the incident light beam for three samples of thermo-tropic polyester fibers of structure (PCPT-co-CPO) with different molar fraction.

fiber with the air field fringes q_s . The number of these positions m_s vary from sample to other sample depending on the sample structure. The values of the interfering spacing b_s decreased with decreasing the wavelength of the light vector incident on the fiber which is the bases of the measurements using VAWI technique. The intensity of illumination of the fringes obeys two beam interference fringes which characterized by bright and dark fringes of equal width. The relation between the relative intensity distribution of and the wavelength of the incident light beam was plotted, using eq. (1), as shown in Figure 2. The number of the maximum and mini-

um peaks for each sample depends on its birefringence. The variation of this number from sample to another denotes to the number of the positions of coincidence and anticoincidence for this sample.

For a quantitative evaluations of the analysis, the birefringence dispersion data were obtained using eqs. (3) and (4). The results of the birefringence and wavelength for each sample, Tables I–III, were extrapolated, using the polynomial best fit, for the range of the wavelengths from 400 to 700 nm. Figure 3 shows the relation between the birefringence and the wavelengths for each sample which gives the spectral dispersion curves of the birefringence of the three samples of thermo-tropic polyester fibers of structure (PCPT-co-CPO) with different molar fraction. It is obvious that, the birefringence values increased with increasing the copolymer disruptor unit concentration of molar fraction [CPO] from 0.40 to 0.60. Although Samples 1 and 3 of copolymer molar fraction [CPO] = 0.40 and [CPO] = 0.60 were made as spun and Sample 2 of copolymer [CPO] = 0.50 was thermally treated, the thermal treatment has no effect on the birefringence value of Sample 2. So that the improvements of the birefringence must be attributed to the rise of the molecular weight. The increase in the birefringence indicates that the thermotropic polyesters fibers changed into more oriented fibers by adding higher values of the molar fractions of copolymer. Consequently, birefringence is expected to be very sensitive to fiber structure on the molecular level.

To verify he Cauchy's dispersion formula, a relation between the birefringence $\Delta n(\lambda)$ and the reciprocal of the square of the wavelength, $1/\lambda^2$, in the wavelength range from 400 to 700 nm was plotted.

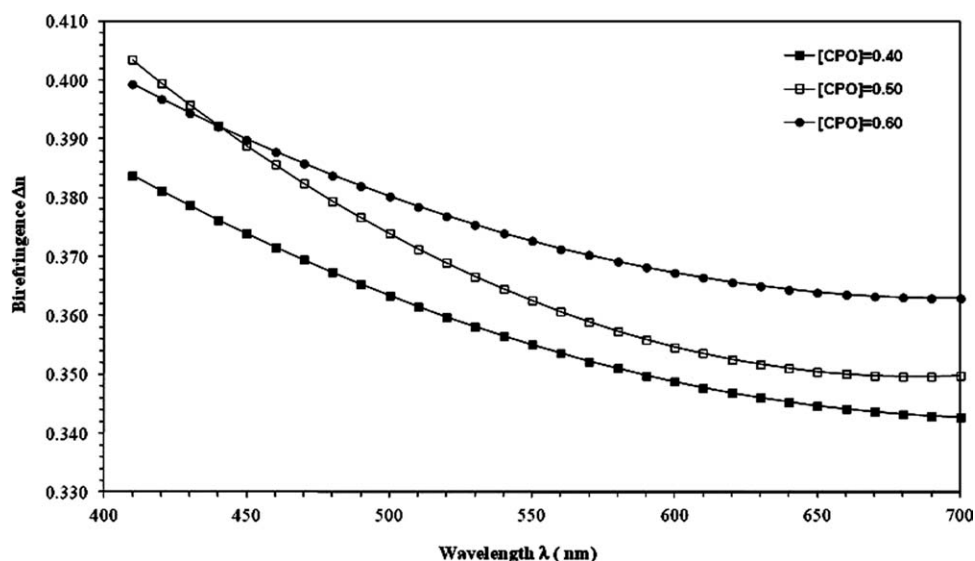


Figure 3 The spectral dispersion curves of the birefringence for the three samples of thermo-tropic polyester fibers of structure (PCPT-co-CPO) with different molar fraction.

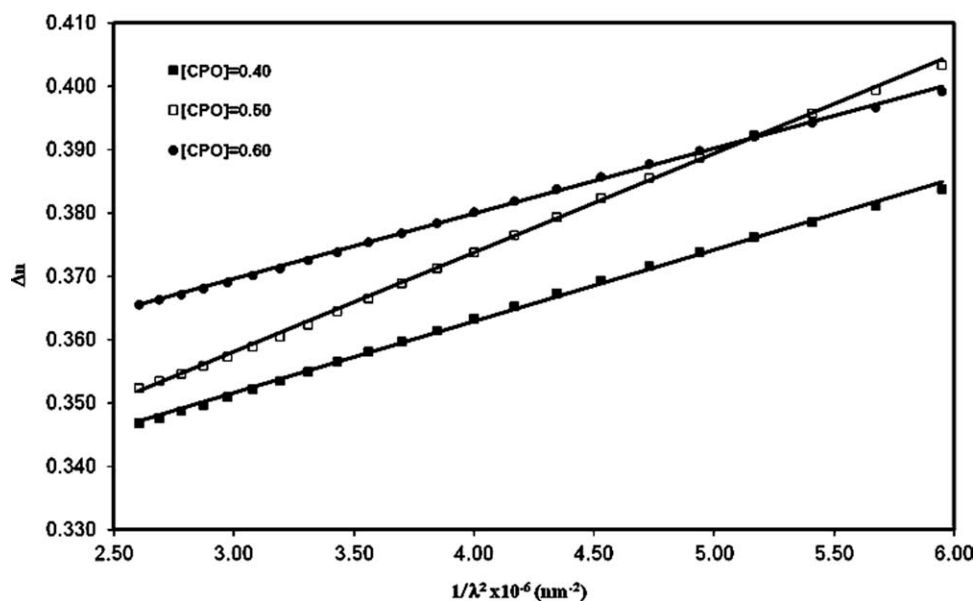


Figure 4 The birefringence Δn as a function $1/\lambda^2$ to verify the Cauchy's dispersion formula for the three samples of thermo-tropic polyester fibers of structure (PCPT-co-CPO) with different molar fraction.

A straight line was obtained for each sample, as shown in Figure 4. From the slope and intercept of these lines, the constants ΔA and ΔB of Cauchy's dispersion formula were calculated. The results are given in Table IV. The value of the constant ΔA increased with increasing the molar fractions concentration while the constant ΔB decreased. These constants refer to the high molecular orientation of thermo-tropic polyesters of structure (PCPT-co-CPO) with different molar fraction. They play an important role in determining the behavior of the birefringence of thermo-tropic polyesters and properly normalize the interaction potential describing the optical effects.

The birefringence profiles of thermo-tropic polyester fibers using VAWI-technique

Also, for a quantitative evaluations of the analysis, a mathematical expression for determining the birefringence profile was derived. The analysis is depending on the method previously described by Sokkar and El-Bakary¹⁶ for highly oriented fibers

TABLE IV
Results of the Cauchy's Dispersion Constants For the Birefringence of Three Samples of Thermo-Tropic Polyester Fiber of Structure (PCPT-co-CPO) with Different Molar Fraction Using VAWI Technique

Samples	ΔA	$\Delta B \times 10^{-3}$ (nm^2)
Sample 1 of copolymer [CPO] = 0.40	0.3111	15.6
Sample 2 of copolymer [CPO] = 0.50	0.3176	11.3
Sample 3 of copolymer [CPO] = 0.60	0.3387	10.3

using the VAWI technique. The method depends initially on measuring the fringe shift profile at a certain coincidence position, then the birefringence profile can be measured. To determine the fringe shift profile we choose an available fringe shift at a coincidence position of the fiber with the air fringes that is seen in the field of the microscope. The number of interference order m_s was measured and hence the distance $m_s b_s$ from the zero order fringes was calculated. The fringe shift was divided into layers and the distance under the fringe shift Z_x was measured at each layer. The available values of the distances Z_x covers about 70% of the fiber diameter. A numerical method, as a polynomial best fit was used to determine the complete shape of the fringe shift profile. The wavelength of light used was 613.5 nm at coincidence position for each sample. Figure 5 shows the fitted fringe shift profile for three samples of thermo-tropic polyesters of structure (PCPT-co-CPO) with different molar fraction. The best fitted data for the distances Z_x for each sample was used with eq. (8) to calculate the birefringence profile for the three samples of thermo-tropic polyester of structure (PCPT-co-CPO) with different molar fraction. The graphical representation of the calculations is shown in Figure 6. It is clear from this figure that, all profiles take the same behavior but the difference only in the values of the birefringence of each sample. The birefringence profile for Sample 3 of copolymer [CPO] = 0.60 has greatest birefringence values, Sample 2 of copolymer [CPO] = 0.50 has medium birefringence values, and Sample 1 of copolymer [CPO] = 0.40 has lower birefringence values. The presented data show that the three samples have the same

structure behavior and the changes in copolymer composition ([CPO]) between 0.40 and 0.60 substantially affects the physical properties of PCPT-co-CPO fibers. Hence the observed improvements of birefringence must be attributed to the rise of molecular weight which is agreed with the previously published work.⁵

In most interferometric methods used for determining the birefringence profile, the length of the fringe shift at the fiber center must be less than or equal one interference order. In our method, the length of fringe shifts at the fiber center m_s equals 13 order in case of Sample 1 of copolymer [CPO] = 0.40, $m_s = 14$ in case of Sample 2 of copolymer [CPO] = 0.50 and $m_s = 18$ in case of Sample 3 of copolymer [CPO] = 0.60. This is the main advantage of our measuring method using the VAWI technique.

CONCLUSION

Thermo-tropic polyester fiber of structure (PCPT-co-CPO) is one of most high performance fibers and has remarkable unique interesting properties and potential applications. The VAWI technique is suitable for studying optical anisotropy of such high mechanical strength and high performance fibers. This technique is also particular important for determining the spectral dispersion properties of such of these fibers which characterize the refraction proper-

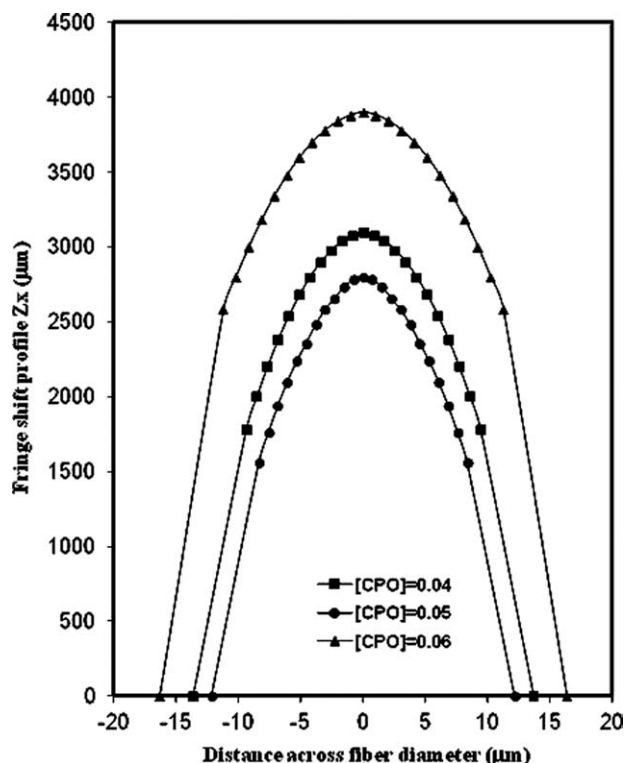


Figure 5 The fringe shift profile for three samples of thermo-tropic Polyester fibers of structure (PCPT-co-CPO) with different molar fraction.

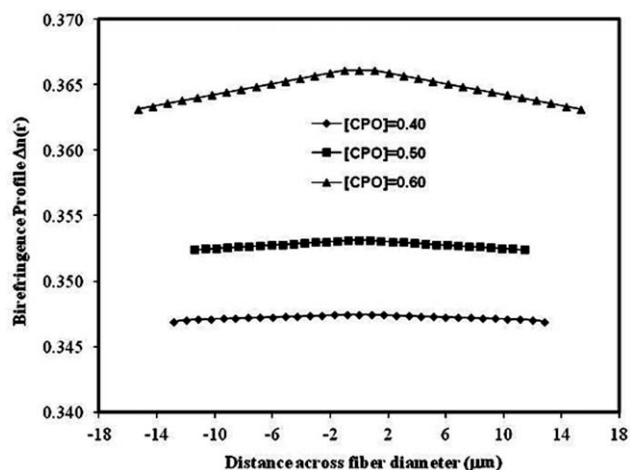


Figure 6 The birefringence profile for the three samples of thermo-tropic polyester fibers of structure (PCPT-co-CPO) with different molar fraction.

ties of thermo-tropic polyester fibers in the visible portion of spectrum. The use of VAWI technique verified the Cauchy's dispersion formula for thermo-tropic polyester of structure (PCPT-co-CPO) and determined its constants. These constants relate the molecular arrangement of these fibers.

A mathematical expression was derived for direct measurement of the birefringence profile, taking the refraction into consideration, of such highly birefringent fibers. The measurement of birefringence profiles, considering the fibers consist of layers, threw light on the internal structure of each layer and detected the variation of these structural properties across the fiber diameter. These profiles play an important role in the development of mechanical properties of thermo-tropic polyester of structure (PCPT-co-CPO) fibers.

The presented data show that the three samples have the same structure behavior and the changes in copolymer composition ([CPO]) between 0.40 and 0.60 substantially affects only the physical properties of PCPT-co-CPO fibers. Hence the observed improvements of birefringence must be attributed to the rise of molecular weight.

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