Influence of Grafting on Structural and Optical Properties of Nylon-6 Fibers

E. A. Seisa

Department of Physics, Faculty of Science, Mansoura University, Mansoura, Egypt

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ABSTRACT: This study throws light on the change of the optical properties and some structural properties due to graft copolymerization of polydiallyldimethyl ammonium chloride (PDADMAC) and polyacrylamide (PAA) of nylon-6 fibers. Multiple-beam interferometric technique in transmission was used to study the change of the diameter, refractive indices, and birefringence of nylon-6 fibers at different graft yields. The results were utilized to investigate the isotropic refractive index, the mean polarizabilities per unit volume, dielectric constant, dielectric susceptibility, and surface reflectivity for nylon-6 and grafted nylon-6 fiber. The effect of grafted PAA onto modified nylon-6 fibers containing PDADMAC on the crystallinity was studied by X-ray diffraction. These results reflect good effect of grafting on the optical and structural properties of nylon-6 fibers. The opto-thermal properties of grafted PAA with different graft yields have been studied. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 117: 3255–3261, 2010

Key words: nylon-6 fiber; grafted nylon-6 fiber; polyacrylamide; interferometry; optical properties; X-ray diffraction

INTRODUCTION

Grafting copolymers of various monomers with synthetic and natural polymer have been widely studied in recent years. The grafting polymerization is a well known method to modify the chemical and physical structure of polymers, and to consequently expanding their application. Several studies have appeared dealing with grafting vinyl monomers onto polymers.[1–5] Many ways were developed to prepare grafting copolymer, such as high energy (gamma ray, electron beam),[6,7] plasma treatment,[8] ultraviolet light (UV),[9,10] chemical initiators (including grafting polymerization in solution and in melt)[11–13] and oxidation of polymer. However, the most common method to prepare grafting copolymer is initiated by chemical initiators.

Synthetic fibers occupy a very important position in textile field. Among these, nylon-6 fibers have an important place because of its set of unique, intrinsic characteristics. However, nylon-6 fibers have some inherent drawbacks, which limit their fields of usage, and there by directed the attention toward improving their properties.

Chemical modification of nylon-6 fibers via grafting with vinyl monomers has gained considerable attention during the last decades. Certain desirable properties such as creation of ion exchange properties improving in dyeability, antistatic properties, and in moisture regain.[14,15]

Interferometry is a very useful tool in the fiber science. Two-and multiple-beam interferometric techniques are accurate and nondestructive techniques in the field of fiber research. The resulting fringes give information about the refractive indices and birefringence of the fiber under investigation.[16–18]

Birefringence is one of the most sensitive indicators of the anisotropy of properties in polymers fibers, i.e., the degree of macromolecular orientation. As birefringence is a measure of the total molecular orientation of the two phase system, its examination in conjunction with other physical measurements (X-ray, density, mechanical loss factor, etc.) yields considerable insight into the characteristic of the bulk polymer.[19]

Application of microscopy to thermal analysis provides the means for identification of fibers. It gives knowledge about all phenomena concomitant with the temperature rise of a substance up to its melting point such as polymorphic transformation, birefringence decomposition, etc.

The object of the work described in this article was to study the effect of graft nylon-6 fibers with polydiallyldimethyl ammonium chloride (PDADMAC) and polyacrylamide (PAA) on the optical, structural, and opto-thermal properties at different graft yields.

Correspondence to: E. A. Seisa (seisa@mans.edu.eg).

THEORETICAL

The mean refractive indices and mean birefringence

Multiple-beam Fizeau fringes in transmission technique were used to determine the mean refractive indices and birefringence of grafted nylon-6 fiber. The following formula was to overcome any irregularity of the fringe shift of the fiber[16]

$$n^{\parallel} = n_L + \frac{F^{\parallel}}{2\mathrm{A}} \cdot \frac{\lambda}{h} \tag{1}$$

with an analogous formula for $n^{\perp}$, where $n^{\parallel}$ and $n^{\perp}$ are the mean refractive indices for the light vibrating parallel and perpendicular to the fibers axis, respectively; n_L is the refractive index of the immersion liquid, $F^{\parallel}$ and $F^{\perp}$ are the total area of fiber under the fringe shift, A is the fiber cross-sectional area, h is the interfringe spacing between any two consecutive bright or dark fringes in liquid region, and λ is the wavelength of monochromatic light used. The mean birefringence $\Delta n = n^{\parallel} - n^{\perp}$, given by the following equation:

$$\Delta n = \frac{(F^{\parallel} - F^{\perp})}{2A} \cdot \frac{\lambda}{h} \tag{2}$$

The polarizability per unit volume

The Polarizability per unit volume can be calculating from the following equation[20]:

$$p^{\parallel} = \left(\frac{3}{4\pi}\right)\left(\frac{n_{\parallel}^2 - 1}{n_{\parallel}^2 + 2}\right) \tag{3}$$

where $p^{\parallel}$ is the polarizability per unit volume in the parallel direction and an analogous equation in the perpendicular direction.

Isotropic refractive index

As most macromolecular crystals are birefringent, an appropriate average refractive index must be used and can be given by the following equation[21]:

$$n_{\mathrm{iso}} = (n^{\perp 2}\, n^{\parallel})^{1/3} \tag{4}$$

Dielectric constant and dielectric susceptibility

The dielectric constant and dieletric susceptibility are the most important parameters that play a major role in the determination of material properties. The dielectric constant is given by the following relation[22]:

$$\varepsilon_{\parallel} = \frac{1 + 2(n_{\parallel}^2 - 1)/(n_{\parallel}^2 + 2)}{1 - (n_{\parallel}^2 - 1)/(n_{\parallel}^2 + 2)} \tag{5}$$

with a similar formula for $\varepsilon_{\perp}$. The dielectric susceptibility η is related to the dielectric constant ε by the well-known equation

$$\eta = \frac{\varepsilon - 1}{4\pi} \tag{6}$$

Calculation of the surface reflectivity

The surface reflectivity of a polymer for light at normal incidence can be estimated from Fresnel's equation[22] and the mean refractive index $\overline{n}$. Thus the reflection $\overline{R}$ is given by,

$$\overline{R} = \left(\frac{\overline{n} - 1}{\overline{n} + 1}\right)^2 \times 100 \tag{7a}$$

The transmittance τ_i can be given from the following equation

$$\tau_i = 1 - \overline{R} \tag{7b}$$

The degree of crystallinity

The degree of crystallinity, χ determined by X-ray diffraction is calculated by the following equation[23]:

$$\chi = \frac{I_c}{I_c + KI_a} \tag{8}$$

where I_c and I_a are the integrating intensities scattered over a suitable angular interval by the crystalline and the amorphous phases, respectively, and K is the calibration constant.

EXPERIMENTAL

Materials

Nylon-6 fibers used throughout this study (210 dynier/135 filaments, density 1.14 g/cm^3) were kindly supplied by Misr Rayon Co. (Kafr El- Dawar, Egypt).

Preparation of nylon-6 fibers containing PDADMAC and PAA was carried out according to the method described by Ahmed.[24] These samples were kindly supplied by Textile Research Division, National Research Center, Dokki, and Cairo, Egypt.

Two groups of samples were used in this work; the first group includes nylon-6 fibers (blank), nylon-6 containing 1, 1.5, and 1.9% of grafted PDADMAC.

Second group includes nylon-6 fibers containing PDADMAC at grafted 1.5% and nylon-6 containing 16, 24, and 40% of grafted PAA onto modified nylon-6 fibers containing PDADMAC 1.5%.

TABLE I
Diameter, Refractive Indices ($n^{\parallel}$, $n^{\perp}$) and Birefringence Δn at Different Graft Yields of Nylon-6 Fibers

Sample	Diameter (μm)	$n^{\parallel}$	$n^{\perp}$	Δn
First group sample				
Nylon-6 (blank %)	43.478	1.5452	1.5243	0.0210
Nylon-6-*g*-PDADMAC (1%)	45.455	1.5480	1.5240	0.0240
Nylon-6-*g*-PDADMAC (1.5%)	47.431	1.5490	1.5223	0.0266
Nylon-6-*g*-PDADMAC (1.9%)	48.419	1.5493	1.5221	0.0273
Second group sample				
Nylon-6-*g*-PDADMAC (1.5%)	47.431	1.5490	1.5223	0.0266
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%)	49.407	1.5548	1.5204	0.0344
Nylon-6-*g*-PDADMAC (1.5%)-*g*- PAA (24%)	51.383	1.5555	1.5181	0.0374
Nylon-6-*g*-PDADMAC (1.5%)-*g*- PAA (40%)	53.360	1.5559	1.5179	0.0380

Measuring technique

Optical systems

The studies were carried out using an automatic multiple-beam Fizeau interferometery[16] to find out the variation of the diameter, refractive indices, and birefringence of nylon-6 and grafted nylon-6 fiber at different graft yields.

The Pluta interference microscope[25,26] connected to opto-thermal device[27] was used to study the temperature effect on optical properties of grafted nylon-6 fibers.

The obtained fiber image (microinterferogram) was captured using a charged coupling device (CCD) camera to determine the diameter of fibers and the principle optical parameters of graft nylon-6 fibers.

X-ray diffraction

X-ray diffraction (XRD) studies were carried out using a Shimadzu (Dx 30) X-ray diffractometer, with Cu target and Ni filter were used at 4.5 KV and 35 mA. All diffraction patterns were recorded, supplying radiation of wavelength 0.15406 nm in a wide range from $3.74^{\circ} < 2\theta < 59.77^{\circ}$ with a step interval of 0.02°.

RESULTS AND DISCUSSION

In this work, we report on the results of graft copolymerization of PDADMAC and PAA onto nylon-6 fibers. Moreover, diameter, mean refractive indices, the mean birefringence, isotropic refractive index, the polarizabilities per unit volume, dielectric constant, dielectric susceptibilities, and surface reflectivity of parent and grafted fibers were evaluated.

Measurement of diameter of grafted nylon-6 fiber

The diameter of grafted nylon-6 fiber has been measured by high power optical microscope. The change in the diameters of nylon-6 fiber with different

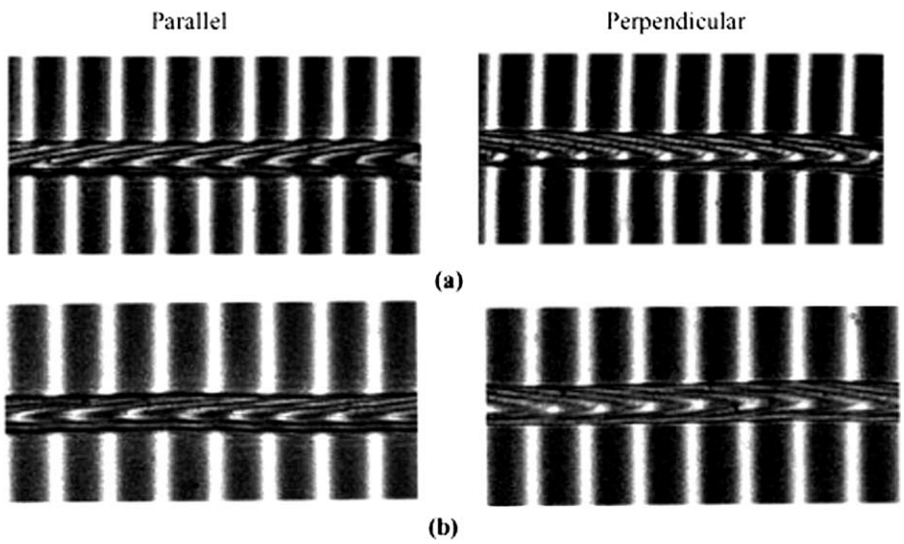

Figure 1 Microinterferogrames of multiple-beam Fizeau fringes in transmission for light vibrating parallel and perpendicular to the fiber axis; (a) Nylon-6 fiber (blank). (b) Nylon-6-*g*-PDADMAC (1.9%).

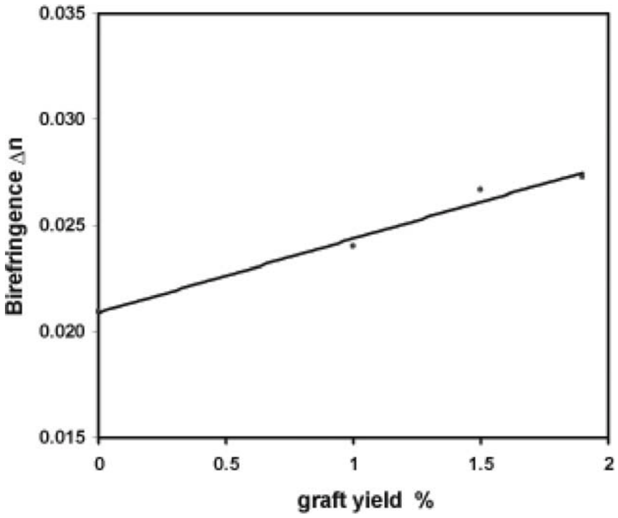

Figure 2 The relation between the mean birefringence Δn of nylon-6 fibers with different graft yields % (for the first group of samples).

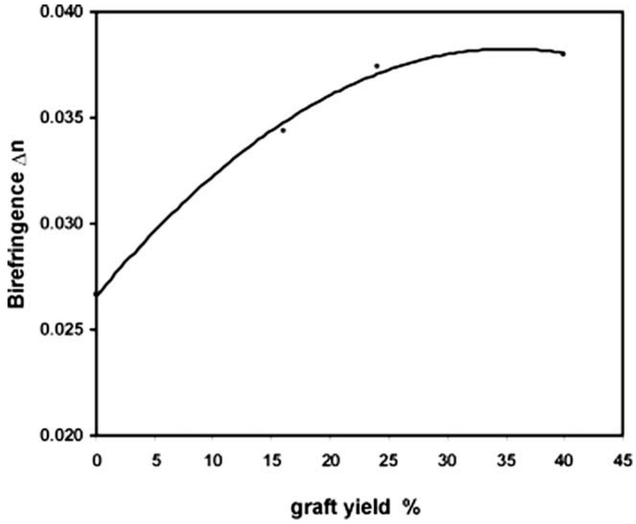

Figure 4 The relation between the mean birefringence Δn of nylon-6 fibers with different graft yields % (for the second group of samples).

grafting yields (for two groups) was shown in Table I. It was found that the fiber diameter increases as grafting yield increasing.

Graft of PDADMAC onto nylon-6 fibers

Multiple-beam Fizeau fringes in transmission method were used to study the changes in optical properties due to grafting of nylon-6 fibers with PDADMAC with different graft yields (1, 1.5, and 1.9%).

Figure 1(a,b) microinterferograms of multiple-beam Fizeau fringes in transmission for [nylon-6 (blank)] and [nylon-6-*g*-PDADMAC (1.9%)] for light vibrating parallel and perpendicular to the fibers axis. Monochromatic light of wavelength 546.1 nm was used and refractive index of the immersion liquid was 1.536 at 19°C. The mean refractive indices ($n^{\parallel}$, $n^{\perp}$) and the mean birefringence Δn are calculated using the microinterferograms and eqs. (1) and (2). The results of first group are given in Table I.

Figure 2 shows the relationship between the mean birefringence Δn with different graft yields for first group of samples. This indicates that the birefringence is increase linearly with increasing the graft yield.

Graft PAA onto modified nylon-6 fibers

The changes of the refractive indices and birefringence have been measured by multiple-beam Fizeau technique due to grafted PAA onto modified nylon-

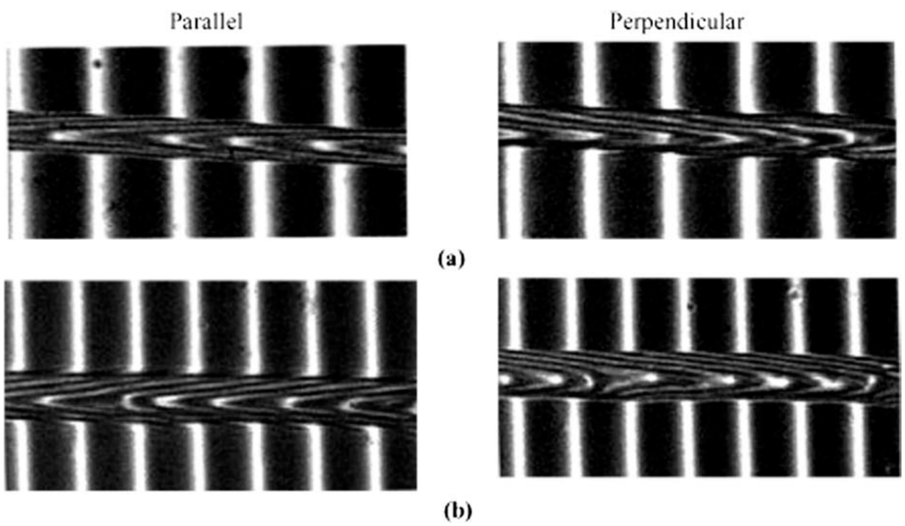

Figure 3 Microinterferogrames of multiple-beam Fizeau fringes in transmission for light vibrating parallel and perpendicular to the fibre axis; (a) Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%). (b) Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (40%).

TABLE II
Isotropic Refractive Index n_{iso}, Polarizabilities ($p^{\parallel}$, $p^{\perp}$), Mean Polarizability ΔP, Dielectric Constant ($\varepsilon^{\parallel}$, $\varepsilon^{\perp}$), Dielectric Susceptibility ($\eta^{\parallel}$, $\eta^{\perp}$) and Surface Reflectivity at Different Graft Yields of Nylon-6 Fibers

Sample	n_{iso}	$p^{\parallel}$	$p^{\perp}$	ΔP	$\varepsilon^{\parallel}$	$\varepsilon^{\perp}$	$\eta^{\parallel}$	$\eta^{\perp}$	$\overline{R}$
First group sample									
Nylon-6 (blank %)	1.5312	0.0755	0.0731	0.0024	2.3877	2.3234	0.1104	0.1053	4.4049
Nylon-6-*g*-PDADMAC (1%)	1.5320	0.0758	0.0730	0.0028	2.3963	2.3226	0.1111	0.1052	4.4146
Nylon-6-*g*-PDADMAC (1.5%)	1.5311	0.0759	0.0728	0.0031	2.3993	2.3174	0.1113	0.1048	4.4040
Nylon-6-*g*-PDADMAC (1.9%)	1.5311	0.0760	0.0728	0.0032	2.4004	2.3167	0.1114	0.1046	4.4035
Second group sample									
Nylon-6-g-PDADMAC (1.5%)	1.5311	0.0759	0.0728	0.0031	2.3993	2.3174	0.1113	0.1048	4.4040
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%)	1.5317	0.0766	0.0726	0.0040	2.4173	2.3115	0.1128	0.1044	4.4124
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (24%)	1.5304	0.0767	0.0723	0.0043	2.4196	2.3045	0.1130	0.1038	4.3955
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (40%)	1.5305	0.0767	0.0723	0.0044	2.4208	2.3041	0.1131	0.1038	4.396

6 fibers containing PDADMAC (1.5%) with different graft yields (16, 24, and 40%).

Figure 3(a,b) shows the microinterferograms of multiple-beam Fizeau fringes in transmission for [nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%)] and [nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (40%)] for light vibrating parallel and perpendicular to the fibers axis. The results of second group were given in Table I.

Figure 4 shows the relationship between mean birefringence of nylon-6 fibers with different graft yields for the second group of samples. From this figure, it shows that an increase in birefringence with increasing the amount of grafted PAA onto modified nylon-6 fibers. This means that more orientation occur involving molecular arrangements in both crystalline and in amorphous regions. The results of the refractive indices for light vibrating in the directions parallel and perpendicular to the fiber axis were used to calculate the isotropic refractive index n_{iso}, the polarizabilities per unit volume ($p^{\parallel}$ and $p^{\perp}$), dielectric constant ($\varepsilon^{\parallel}$ and $\varepsilon^{\perp}$), dielectric susceptibilities($\eta^{\parallel}$ and $\eta^{\perp}$), and surface reflectivity $\overline{R}$ (for two groups) are given in Table II. It is apparent from Table II that $p^{\parallel}$, $\varepsilon^{\parallel}$, and $\eta^{\parallel}$ increase but $p^{\perp}$, $\varepsilon^{\perp}$, and $\eta^{\perp}$ decrease with increasing the amount of grafted yield. The obtained data confirms the applicability of refractive indices for nylon-6 fibers.

X-ray diffraction

The application of XRD technique was used in this work to determine the crystallinity structure of the nylon-6 and graft PAA onto modified nylon-6 fibers as shown in Figure 5. It is clear that the degree of crystallinity of the modified nylon-6 fibers is found to be less than that of the parent one. This effect may be due to the grafting PAA on modified nylon-6 fibers and the decreasing trend depends on the amount of the grafted PAA. According to the matrix effect, we can assume that the grafted surface layers in the earlier stages of the grafting process, in contact with highly oriented surface layers of the nylon-6 matrix, will also have a certain degree of orientation. Table III shows the effect of the amount grafted PAA on crystallinity of nylon-6 fibers. This effect decreases with increasing PAA add-on; the results are in good agreement with that previously in Ref. 4.

Influence of temperature of grafted nylon-6 fibers

The effect of temperature on the optical properties of grafting PAA onto modified nylon-6 fibers containing PDADMAC (1.5%) with different graft yields (16%, 24% and 40%); this throws light on the opto-thermal

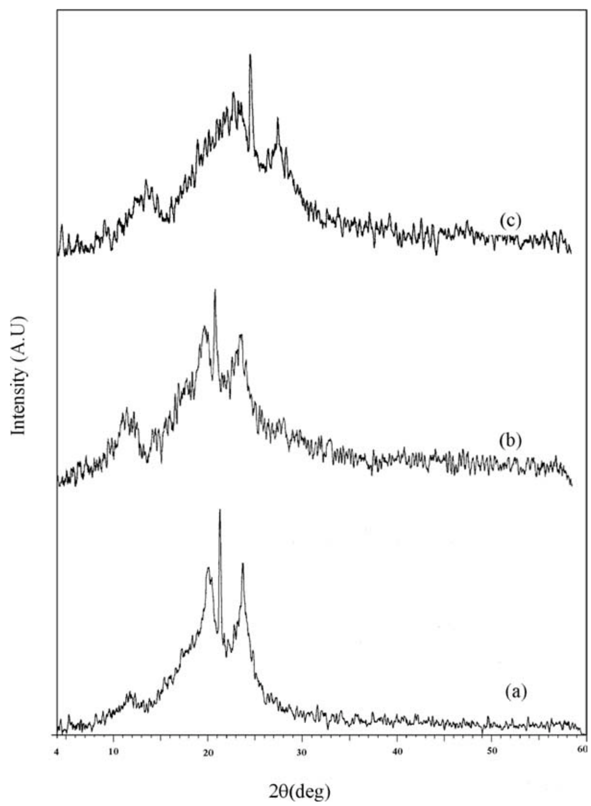

Figure 5 X-ray diffraction pattern for (a) Nylon-6 (blank), (b) Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%), and (c) Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (40%).

TABLE III
Effect of the Amount of Grafted PAA on Crystallinity of Nylon-6 Fibers

Sample	2θ value			Crystallinity (%)
	1st peak	2nd peak	3rd peak	
Nylon-6 (blank)	20.027	21.297	23.719	38.11
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%)	20.18	21.273	24.021	28.00
Nylon-6-*g*-PDADMAC (1.5%)-*g*- PAA (40%)	19.777	21.339	23.685	19.35

behavior of the grafted fibers. The investigated fiber was placed on slide and placed on the Pluta microscope stage connected to opto-thermal device. The temperature controller was used to adjust the temperature of the experiment in the range (27.5–65°C). The microscope was adjusted in a position for producing duplicated images, where the two fringe shifts for light vibrating parallel and perpendicular to the fiber axis. The refractive index of the immersion liquid was 1.5435 at room temperature. Using an Abbe refractometer, the thermal coefficient of the refractive indices of the immersion liquids were determined and found to be ($3.75 \times 10^{-4\circ}C^{-1}$).

Figure 6(a,b) shows the microinterferograms of the totally duplicated image from Pluta interference microscope for light vibrating parallel and perpendicular to the fiber axis, respectively. Figure 7 illustrates the variation of the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) with the increasing temperatures for the aforementioned samples. Overall orientation and the alignment chains in parallel and perpendicular directions of the graft nylon-6, are affected by increasing the temperature. The results obtained for grafted nylon-6 indicate that the refractive indices ($n^{\parallel}$ and $n^{\perp}$) decrease linearly with increasing temperature. The opto-thermal coefficients of refractive indices ($n^{\parallel}$ and $n^{\perp}$) were calculated for samples and listed in Table IV. Figure 8 shows the relationship between the mean polarizabilities ($p^{\parallel}$ and $p^{\perp}$) and the temperature for the tested samples. It is clear that the polarizabilities ($p^{\parallel}$ and $p^{\perp}$) decrease linearly with increasing temperature. Measurements of the mean refractive indices and the mean polarizabilities per unit volume give quantitative information about molecular orientation in the grafted nylon-6 fibers.

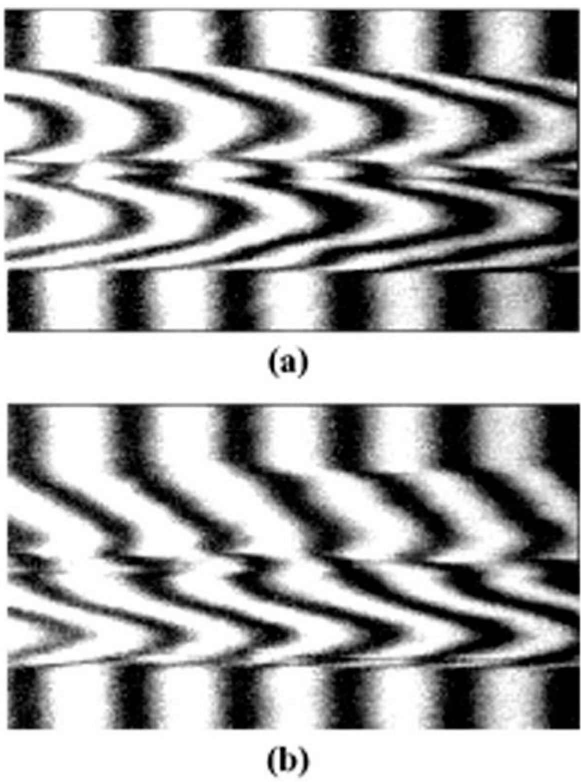

Figure 6 Microinterferograms of the totally duplicated image from Pluta interference microscope for light vibrating parallel and perpendicular to the fiber axis; **(a)** Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%). **(b)** Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (40%).

CONCLUSION

Based on the obtained data one can conclude the following:

1. Fiber diameter increase as grafting yield increases. This may attributed to the possible accumulation of grafted chains in between the polymer chains.[28]

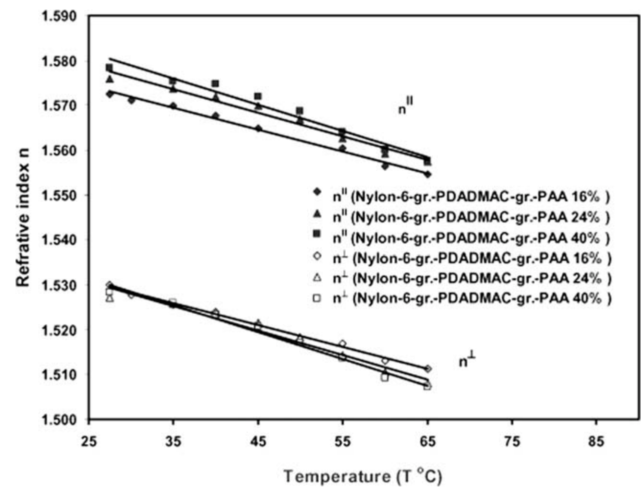

Figure 7 The relation between temperature and the refractive indices $n^{\parallel}$ and $n^{\perp}$ of nylon-6 fibers, containing different amounts of grafted polymer (for the second group of samples).

TABLE IV
The Optical Thermal Coefficient of the Grafted PAA of Nylon-6 Fiber

Sample	$dn^{\parallel}/dT$ (°C^{-1})	$dn^{\perp}/dT$ (°C^{-1})
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (16%)	4.85×10^{-4}	4.86×10^{-4}
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (24%)	5.30×10^{-4}	5.47×10^{-4}
Nylon-6-*g*-PDADMAC (1.5%)-*g*-PAA (40%)	5.87×10^{-4}	6.01×10^{-4}

2. Grafting of polyacrylamide PAA onto modified nylon-6 fibers containing 1.5% of PDADMAC. The effect increases the birefringence and the mean polarizabilities per unit volume with increasing the amount of grafted polyacrylamide. This means an increase in orientation due to different graft levels. The orientation was attributed to that molecular arrangement in both crystalline and amorphous regions had occurred.
3. The degree of crystallinity for the graft nylon-6 fibers is decreased with increasing the amount of grafted PAA. According to the matrix effect, we can assume that the grafted surface layers in the earlier stages of the grafting process, in contact with highly oriented surface layers of the nylon-6 matrix, will also have a certain degree of orientation. This effect decreases with increasing PAA add-on.
4. Increasing the temperature of grafted PAA onto modified nylon-6 fibers containing PDADMAC, the mean refractive indices ($n^{\parallel}$ and $n^{\perp}$) and the mean polarizabilities per unit volume ($p^{\parallel}$ and $p^{\perp}$) decrease. This is observed irrespective of the amount of grafted PAA onto fibers.

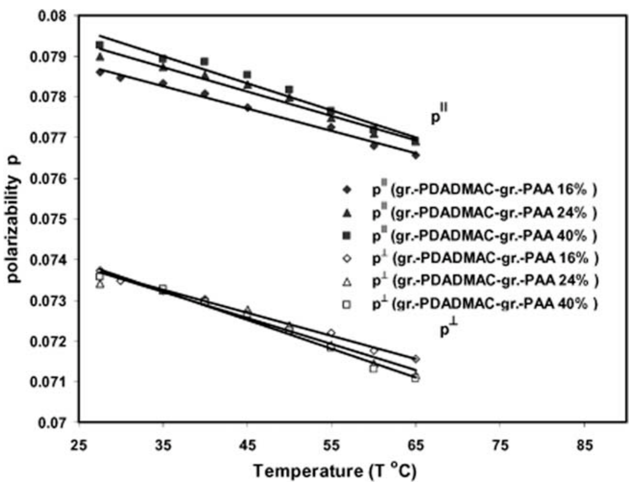

Figure 8 The relation between temperature and the polarizabilities $p^{\parallel}$ and $p^{\perp}$ of nylon-6 fibers, containing different amounts of grafted polymer (for the second group of samples).

We conclude from the previous results that grafting nylon-6 fibers containing PDADMAC and PAA leads to the formation of new structure. These results reflect good effect of grafting on the optical and structural properties of nylon-6 fibers.

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