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CORRELATION OF THE MOLECULAR ORIENTATION AND PHOTONIC PROPERTIES OF RIGID-FLEXIBLE AROMATIC POLYETHERS USING FT-IR LINEAR DICHROISM AND PHOTOLUMINESCENCE SPECTROSCOPIC TECHNIQUES

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**CORRELATION OF THE MOLECULAR
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ABSTRACT

Polymeric films of two types of rigid-flexible luminescent polyethers containing alkyloxy substituted quinquephenyl units and anthracene segments have been uniaxially drawn at temperatures above their glass transition (T_g). FT-IR linear dichroism measurements have been performed in order to evaluate the molecular orientation of the drawn polymers. It was found that the main chain of these polyethers orient parallel to the draw direction. In addition, both the main chain and the side chain for the alkyloxy substituted polyethers also orient parallel to the direction of stress, based on the analysis of the behavior of the ether groups. The concurrent polarized photoluminescence measurements confirm that the high degree of molecular orientation produces an enhancement of the dichroic ratio of the photoluminescence spectra which results in materials with polarized blue and yellow light emission.

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Key Words: Photonic; Infrared linear dichroism; Photoluminescence; Polyethers; Orientation

INTRODUCTION

The understanding of the relationship between the molecular orientation and the properties of polymeric materials is fundamental in processing issues and the long term technological potential of these materials.^[1,2] There are several ways to achieve orientation in polymers and a variety of characterization methods can be used to quantify this very important property.^[1–11] These ways include stretching of the polymer films,^[12–14] deposition on oriented substrates,^[15–19] or by using the liquid crystallinity behavior.^[20–22]

In this work, we have studied the correlation of the molecular orientation of two classes of conjugated polymers as it relates to their photonic properties. Conjugated polymers is a very promising class of materials with unique electro-optical properties.^[23] The use of these materials as displays and other opto-electronic devices depends in a great deal on the ability to orient them using standard polymer processing techniques.^[19,20,24,25] In fact, the strategy to synthesize these materials is governed by issues related to the understanding of the structure-property relationships.^[6] Various types of conjugated units have been used over the years in the creation of rigid-flexible polymers with desirable properties. Such units include phenylene-vinylene,^[27] oligothiophene,^[28] oligophenylene,^[29,30] and bis(styryl)anthracene.^[31] In particular, aromatic-aliphatic polyethers containing either alkyloxy-substituted quinquephenyl units or bistyryl-anthracenes have been synthesized.^[30,31] These polymers exhibit very good solubility and film-forming capability, both properties that are essential to possible technological applications. In addition to these properties, these materials exhibit either blue or yellow light emission, a very important property for display and sensor applications. It is expected that linearly polarized light emission should occur from the photonic excitation of uniaxially stretched films of these kinds of materials.

Therefore, the aim of this study is to determine the molecular orientation of films of aromatic-aliphatic polyethers bearing a) alkyloxy substituted quinquephenyl units and b) anthracene segments using the technique of Fourier transform infrared (FT-IR) Linear Dichroism and to correlate the knowledge gained from the FT-IR experiments to their photonic behavior.^[32,33] A previous study using Raman spectroscopy has shown that rigid-flexible luminescent polyethers show a high degree of molecular orientation and a relatively low dichroic ratio for the photoluminescence measurements.^[34] We expand on this study by using a complementary technique to measure

molecular orientation for the polyether compounds and we examine for the first time the photonic behavior of the anthracene containing rigid-flexible polymer class.

EXPERIMENTAL

Materials

The polymer structures are schematically shown in Fig. 1. The details of the synthesis of the aromatic aliphatic polyethers as well as the conjugated anthracenes used in this study have been published elsewhere.^[30,31] Self-supported films were obtained by melt pressing in a temperature range of 200–210°C for a period of 30 s. Uniaxially oriented films were obtained by drawing the films in a thermostated chamber at temperatures 20–30°C above their glass transition (T_g). The standard drawing speed was ca. 2 cm/min, and the dimensions of the specimen was 2 cm in length, 0.3 cm in width, and 0.01 cm in thickness. These compounds were previously characterized using a variety of analytical techniques that include $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, Raman spectroscopy and FT-IR spectroscopy.

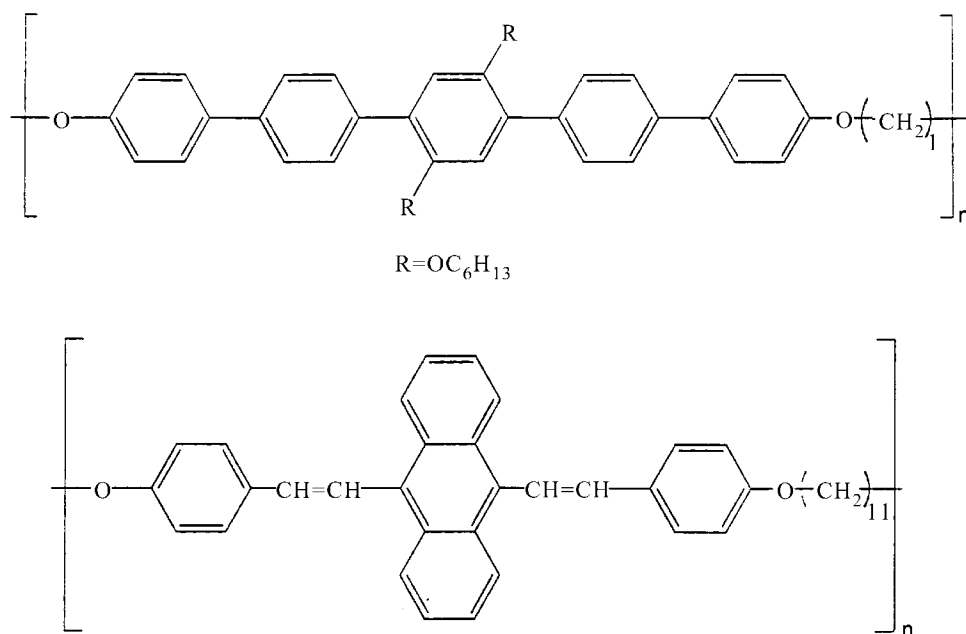


Figure 1. Molecular Structures of polyethers containing a) alkyloxy substituted quinquenyl units (PQP₁) and b) anthracene units in the main chain (PStAn).

FT-IR Linear Dichroism

Infrared dichroism is a tool often used to measure the anisotropy in the orientation of a functional group within a sample. This anisotropy can be caused by either processing conditions, or by the application of external perturbations on the sample (e.g., temperature, mechanical fields or electrical fields).

The static infrared spectroscopic data were collected on a Nicolet 850 Series II FT-IR spectrometer equipped with an MCT/A detector at 2 cm^{-1} spectral resolution. The infrared dichroism studies were performed using a wire-grid infrared polarizer KRS-5 (SPECAC, Kent, UK) that allows plane polarized light to reach the sample. Both free standing polyether films are too thick to be within the range of the Beer-Lambert Law, and saturate many vibrational bands in the transmission infrared spectra. Therefore, the overtones of key fundamental vibrations were used in addition to low intensity fundamentals in the study of the orientation of the different parts of these macromolecules. Overall, the selection of these bands allowed the complete elucidation of the molecular orientation mechanism in these materials.

The use of infrared dichroism involves the orientation of the samples and the subsequent measurement of the absorbance of key vibrational bands parallel and perpendicular to the orientation direction. The dichroic ratio R is defined as $R = \frac{A_{\parallel}}{A_{\perp}}$ where A_{\parallel} and A_{\perp} are the infrared absorbances measured with the radiation polarized parallel and perpendicular to the stretching direction. The value of R can range from zero (where there is no absorption in the parallel direction) to infinity (no absorption in the perpendicular direction). For random orientation, $R = 1$. Another parameter which is very helpful in the determination of the molecular orientation is the so-called order parameter S . The order parameter is derived from the experimentally determined dichroic ratio R ; it is a measure of the orientation of the molecule or the molecular segment. In this study, we have used vibrational bands that are either parallel or perpendicular to the molecular axis, therefore we have used the value of R as the determining quantity in the measurement of the molecular orientation.

In the experiments reported here, a polarizer placed parallel to the direction of stretch was placed between the sample and the detector. The same procedure was carried out on the same sample with the polarizer perpendicular to the stretch direction. Finally, the ratio of the parallel to perpendicular absorbance at each strain value was calculated.

Photoluminescence

Polarized photoluminescence measurement was obtained on a SPEX Fluorolog (Model F212, Spex USA) double monochromator, using polarized

light ($\lambda_0 = 350$ nm) for excitation and a Glan Thomson polarizer on the detector (PMT R928, Hamamatsu Japan). The letter H on the photoluminescence spectra denotes horizontal direction whereas the letter V corresponds to vertical orientation. In all cases, the sample was placed in a vertical orientation (V). For instance, the sequence HVH corresponds to an experimental set-up where the excitation radiation is horizontal, the sample is placed in a vertical way and the analyzed radiation is also horizontal.

RESULTS AND DISCUSSION

Polyether Assignments

Figure 2 shows a typical FT-IR spectrum of the PQP₁ polyether compound before uniaxial drawing. This spectrum is used for reference purposes to show the position of all the fundamental vibrations. As mentioned before, the infrared spectra of the actual polymer films exhibit many spectral regions where saturation occurs, thus making the use of overtones a necessity. Table 1 lists the vibrational assignments for several functional groups that are associated with different parts of the polymer.

Regarding the assignments for the ether vibrations, the infrared bands at 1205 and 1032 cm⁻¹ are assigned to the ether bond of the side chain,^[35] whereas the infrared bands at 1246 and 1054 cm⁻¹ are due to the ether linkage of the main chain.

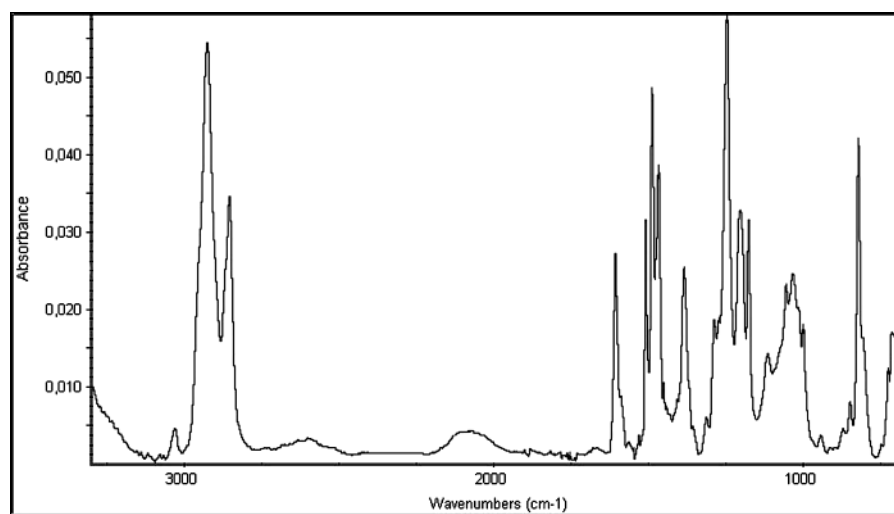


Figure 2. FT-IR spectra of the polyether PQP₁.

Table 1. FT-IR Peak Assignments for PQP₁ Polyether Compound

Wavenumber (cm ⁻¹)	Assignment
3032	CH aromatic stretch vibration
2928	CH ₂ asymmetric stretch vibration
2857	CH ₂ symmetric stretch vibration
2532	C-O-C asymmetric overtone vibration
2424	C-O-C asymmetric overtone vibration
2063	C-O-C symmetric overtone vibration
2023	C-O-C symmetric overtone vibration
1606	C-C aromatic stretch vibration
1488	C-C aromatic stretch vibration
1470	CH ₃ asymmetric deformation
1246	C-O-C asymmetric vibration (main chain)
1205	C-O-C asymmetric vibration (side chain)
1176	CH aromatic in-plane deformation
1054	C-O-C symmetric vibration (main chain)
1032	C-O-C symmetric vibration (side chain)
847	CH aromatic out of plane deformation
821	CH aromatic out of plane deformation
804	CH aromatic out of plane deformation

Molecular Orientation

In order to study the orientation of the main chain and the side chain components of the PQP₁ polyether after uniaxial drawing using the two different polarization geometries mentioned above, the aromatic bands at 3137, 3032, 1915, 1886 and 1579 cm⁻¹, and the ether bands at 2532, 2424, 2063 and 2023 cm⁻¹ were all used. The selection of those particular ether overtone bands was made because of the experimental problems associated with the high thickness of the drawn films that made impossible the calculation of the dichroic ratios of the fundamental ether bands at 1246, 1205, 1054 and 1032 cm⁻¹. Figure 3 shows a typical pair of polarized FT-IR spectra of uniaxially stretched polyether films, at a draw ratio of $\lambda = 1.4$ in this particular case. The draw ratio is defined as the number of times that the original polymer films have been elongated during the stretching process.

The ether bands at 2063, 2023 cm⁻¹ are the overtones of the C-O-C symmetric vibrations, whereas the pair 2532, 2424 cm⁻¹ are the overtones of the C-O-C asymmetric vibrations.

Orientation of the Polyether PQP₁ Main Chain

Figures 4a and 4b show the spectra of the 3032 cm⁻¹ and 1579 cm⁻¹ aromatic infrared bands polarized parallel to the stretch direction at 0, 1.4, 2,

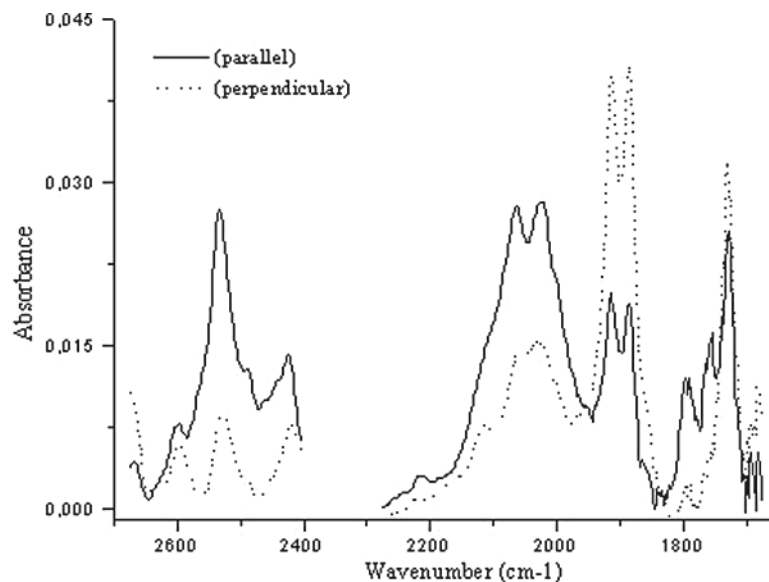


Figure 3. Polarized FT-IR spectra of polyether PQP₁ at draw ratio 1.4.

2.7, 3, 3.8, and 4.7 draw ratios. The 3032 cm^{-1} band corresponds to the CH aromatic stretching vibration, whereas the 1579 cm^{-1} band is associated with the C-C stretching vibration of the aromatic ring. Both bands are parallel polarized and their intensities increase as a function of the draw ratio.

In addition, a small but nevertheless detectable shift of about $1\text{--}2\text{ cm}^{-1}$ wavenumbers for the band maximum is observed for both 3032 and 1579 cm^{-1} bands as we move to higher draw ratios. It has also be noted that the use of the dichroic ratio eliminates problems due to film thinning. The results of the calculation of the dichroic ratios for the 3137 , 3032 , 1579 , 1915 , 1886 cm^{-1} aromatic bands as a function of draw ratio are presented in Fig. 5. Note that there is a residual intrinsic parallel orientation of the samples as seen by the value of R which is higher than 1. Based on these graphs, we conclude that there is a parallel orientation of the main chain of the polyether as we go to higher elongations since the value of R increases monotonically as a function of the draw ratio. Also the behavior of the perpendicularly polarized summation aromatic bands due to the di-substitution of the aromatic ring at 1915 and 1886 cm^{-1} agree with the parallel orientation of the main polymeric chain.

Orientation of Ether Groups

Figures 6a and 6b show the spectra of the 2532 and 2063 , 2023 cm^{-1} ether bands polarized parallel to the stretch direction for the entire series of draw ratio measurements.

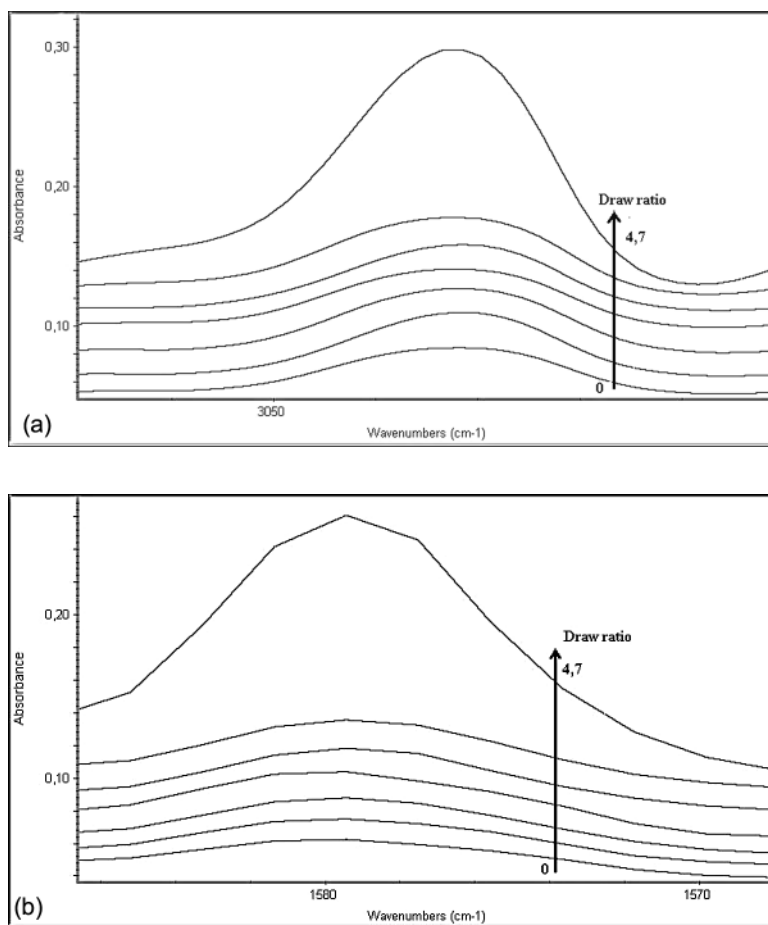


Figure 4. Parallel polarized aromatic bands at 3032 cm^{-1} and 1579 cm^{-1} of polyether PQP_1 at different draw ratios.

The corresponding dichroic ratios for the ether bands as a function of draw ratio are presented in Fig. 7. Based on these calculations, we conclude that both the main as well as the side chain ether functionalities orient parallel to the direction of stress as we move to higher draw ratios. Therefore, the conclusion for the entire macromolecule is that it orients parallel to the direction of stress for high levels of elongation.

Polyether PStAn Assignments

Figure 8 shows the FT-IR spectrum of a thin film of the polyether PStAn bearing anthracene units before any elongation has occurred.

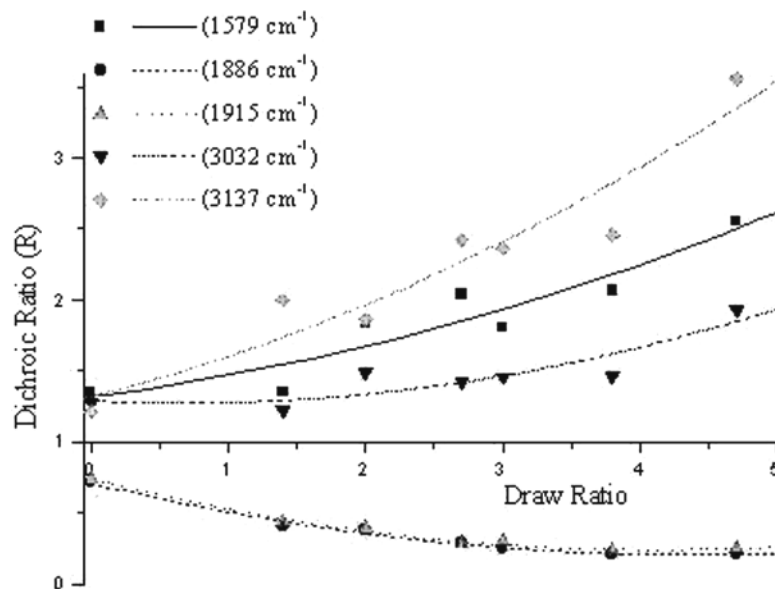


Figure 5. Dichroic ratio of aromatic bands (3137, 3032, 1915, 1886, 1579 cm^{-1}) of polyether PQQ₁ as a function of the draw ratio.

It is obvious that saturation occurs in various spectral regions again due to the high thickness of the materials. For instance, the absorption profile in the region between 1480 and 1430 cm^{-1} shows strongly the effect of saturation. Nevertheless, the existence of several other infrared bands where linearity is obeyed allows us to deduce the molecular orientation of this type of conjugated polymer too. Table 2 depicts the most characteristic peaks and their vibrational assignments of the conjugated polyether containing anthracene units.

Orientation of Polyether PStAn Polymer

In order to examine the direction of the molecular orientation of the main chain of PStnAn, the CH stretching band at 3032 cm^{-1} , which has its origin on the aromatic ring of the main chain, was used. Following the same methodology as before, we find that the main chain of this polymer is oriented parallel to the draw direction as we move to higher elongation lengths. In particular, our experiments resulted in a value of $R = 2.515$ for 3.6 draw ratio.

Orientation of Anthracene Unit

The anthracene specific bands at 1302, 885, 758 cm^{-1} were used to determine the orientation of the anthracene unit itself. It was found that both

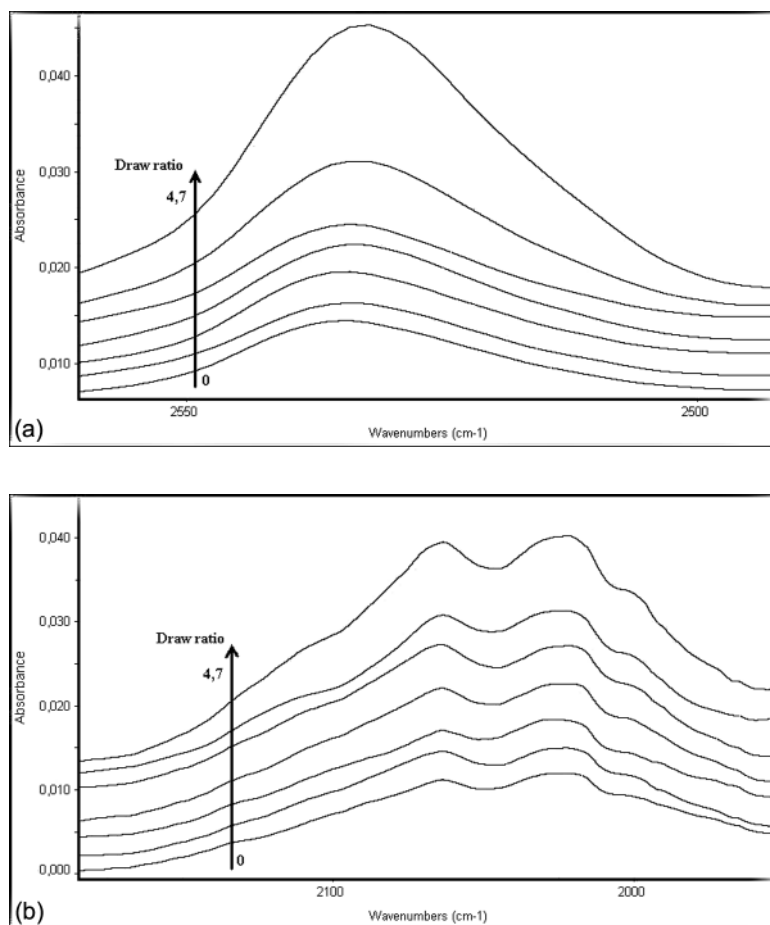


Figure 6. Parallel polarized ether bands at 2532 and 2063, 2023 cm⁻¹ of polyether PQP₁ at different draw ratios.

1302 and 758 cm⁻¹ vibrations exhibit dichroic ratios smaller than unity for 360% elongation, ($R_{1302} = 0.848$, $R_{758} = 0.843$) an indication of a perpendicular orientation, whereas the vibration at 885 cm⁻¹ ($R_{885} = 1.154$) shows parallel orientation. Therefore, we can conclude that the anthracene unit has a perpendicular orientation to the main chain of the polymer.

Luminescence Measurements

The effect of the molecular orientation on the emission properties of these photonic materials becomes evident when we study the photoluminescence spectroscopic results. Both light polarized parallel and perpendicular to the sample have been used for excitation. The dichroic ratios

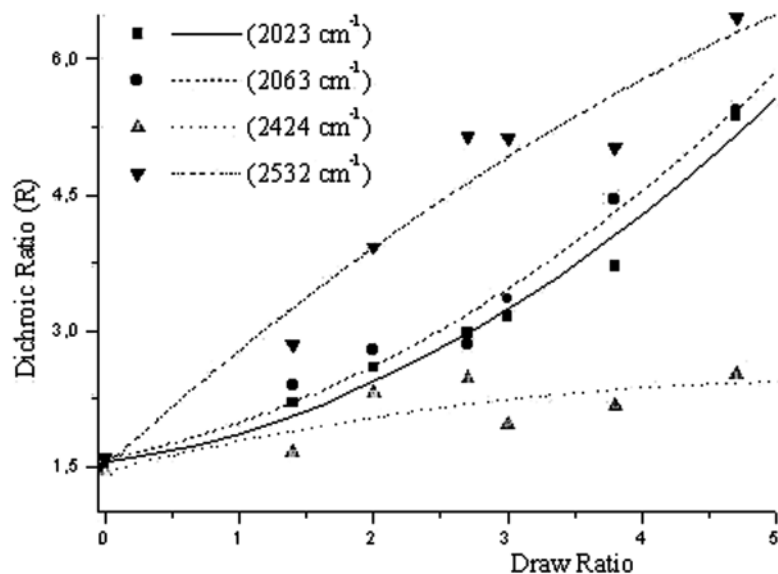


Figure 7. Dichroic ratio of ether bands (2532, 2424, 2063, 2023 cm^{-1}) of polyether PQP₁ as a function of the draw ratio.

were defined and calculated as the ratio between the spectra measured with polarization of the emitted light parallel and perpendicular to the drawing direction. As mentioned in the experimental section H denotes horizontal orientation (perpendicular) whereas V refers to vertical orientation (parallel).

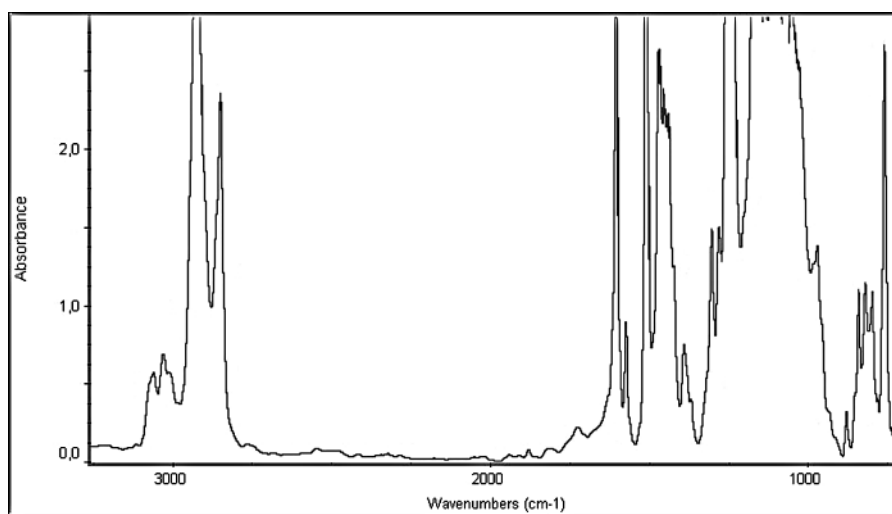


Figure 8. FT-IR spectra of the polyether containing anthracene units (PStAn).

Table 2. FT-IR Characteristic Peaks Assignments in Polyether Containing Anthracene Units (PstAn)

Wavenumber (cm ⁻¹)	Assignment
3032	CH aromatic stretch vibration
2936	CH ₂ asymmetric stretch vibration
2852	CH ₂ symmetric stretch vibration
1605	C-C aromatic stretch vibration
1302	Blending of aromatic C-C-C in plane deformations ³⁶
1258	C-O-C asymmetric vibration
1047	C-O-C symmetric vibration
970	CH double bond bending vibration
885	CH aromatic out-of-plane bending (non adjacent H)
758	CH out-of-plane bending mode of the four adjacent H on the anthracene unit

The integrals rather than the peak maxima have been used since they are directly related to the energy of the relevant electronic transitions.^[37]

The data in Fig. 9 indicate that a small increase in the level of perpendicularly polarized emitted light is observed for the drawn PQP₁ polymer. For instance, the dichroic ratio_{VVV/HVV} = 0.896 and the dichroic

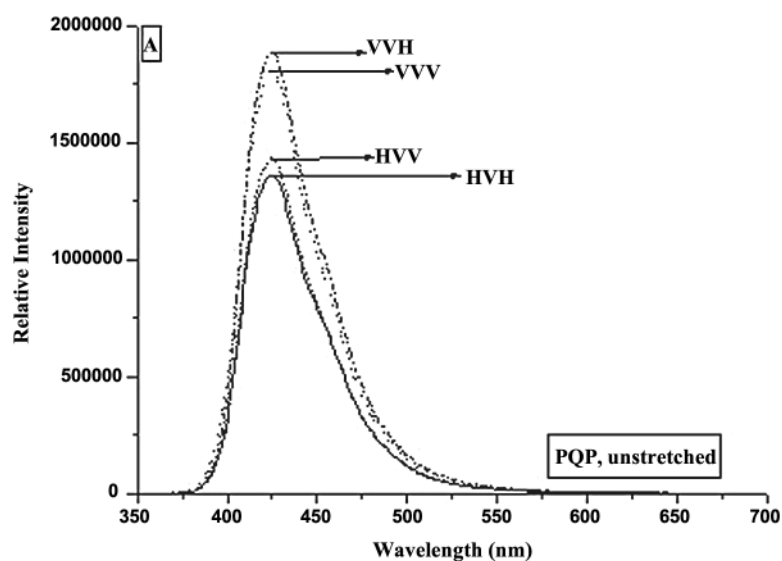


Figure 9. Polarized photoluminescence spectra of polyether PQP₁ A) unstretched film and B) stretched at draw ratio 5.0, and C) absorption-emission profiles in the solid state.

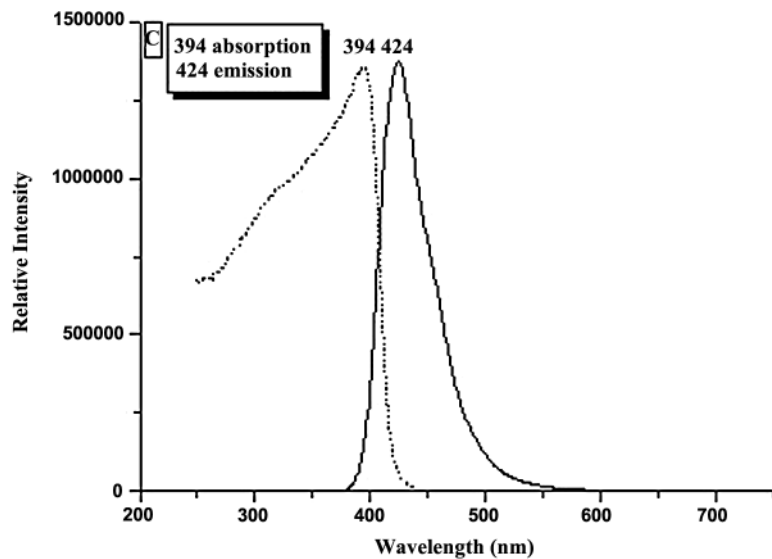
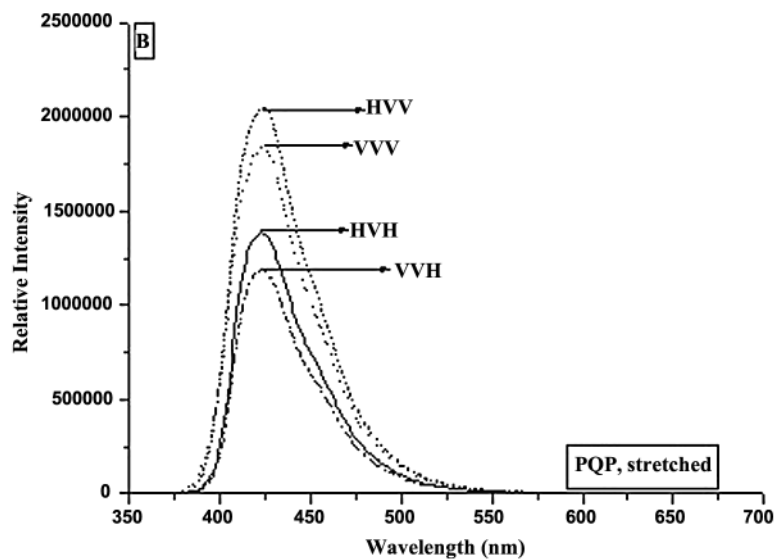


Figure 9. Continued.

ratio $v_{VH}/v_{HV} = 0.857$ for the polymer film stretched 500% its initial length. Surprisingly enough, these observed dichroic ratios for the neat compounds are very low and much lower than the dichroic ratios observed for the infrared measurements. This is in contrast to the high dichroic ratios observed from diluted solutions of similar polymers but in agreement to previous work on neat systems.^[26,34] One possible explanation for this behavior is energy transfer, which can take place in dense systems. The fact that charge carriers

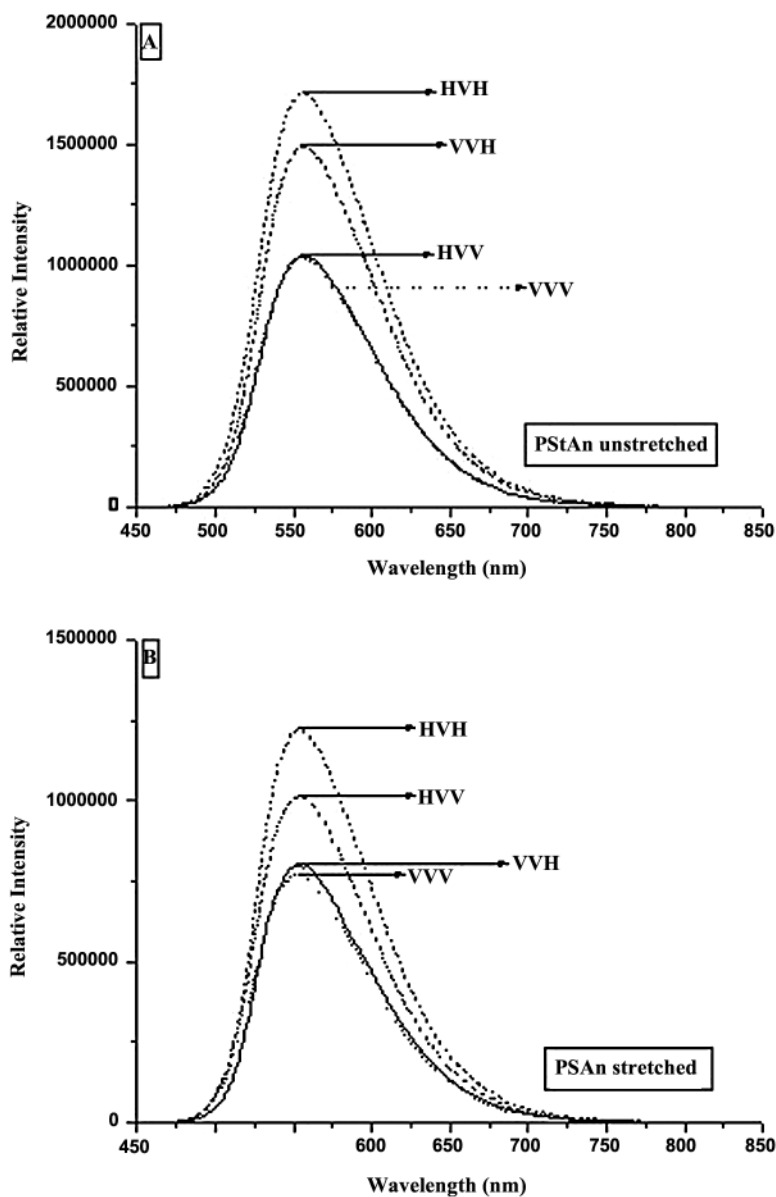


Figure 10. Polarized photoluminescent measurements of polyether PstAn A) unstretched film and B) stretched at draw ratio 3.6, and C) absorption-emission profiles in the solid state.

cannot travel over extend lengths on the main chain has led researchers to consider the mechanism of charge transport via interchain carrier hopping.^[22] Charge transport has become one of the accepted mechanisms for electroluminescent behavior from polymers containing conjugated units

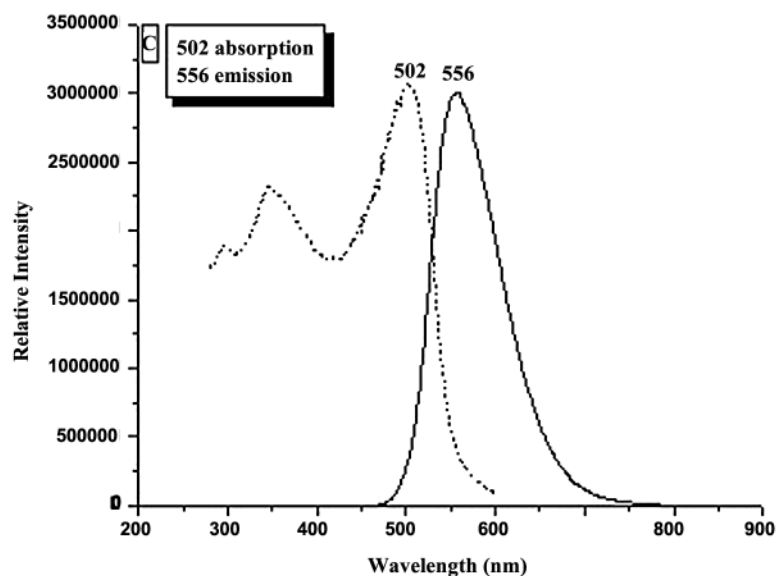


Figure 10. Continued.

and flexible spacers. In particular, charge transfer contributes to the lateral, with respect to molecular orientation, light emission and can be used to explain the data shown in Fig. 9.

In addition, the results of the photoluminescent behavior of stretched vs. unstretched polyether PStAn, depicted in Fig. 10, show that the majority of the polarized emission comes from the anthracene part of the molecule which is oriented perpendicular to the stress direction. Upon elongation of 360%, a decrease of the dichroic ratios VVH/HVH and VVV/HVV is observed (dichroic ratio $_{VVV/HVV} = 0.757$ and the dichroic ratio $_{VVH/HVH} = 0.653$), an indication of the enhancement of the perpendicular yellow light emission.

CONCLUSION

We have clearly demonstrated using FT-IR linear dichroism that both the main and side chain moieties of alkyloxy-substituted aromatic polyether polymers orient parallel to the direction of the externally applied mechanical field. In addition, the main chain of polyethers containing anthracene units also orients parallel to the draw direction, resulting in a conformation having the anthracene unit oriented perpendicular to the stress direction. The concurrent polarized photoluminescence measurements confirm that the high degree of molecular orientation produces an enhancement of the dichroic ratio of the photoluminescence spectra which results in materials with polarized yellow light emission.

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