Spectroscopic Characterization of Plastic Optical Fibers Doped With Fluorene Oligomers

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Abstract—The optical properties of four fluorene oligomers embedded in plastic optical fiber have been investigated by using steady-state luminescence technique. In particular, we study the dependence of the optical spectra with dopant oligofluorene, emission wavelength, excitation wavelength, and light propagation distance through the fiber. Using this information, we have characterized the optical loss in these doped optical fibers.

Index Terms—Dyes, fluorescence, optical fiber amplifiers, optical fiber lasers, optical fiber materials, plastic optical fiber (POF).

I. INTRODUCTION

WITH the development of polymer optical fibers (POFs) in the last years [1], increased research activities have also been carried out in the field of active POF amplifiers and lasers. POF amplifiers that generate signal light in the visible and near-infrared are potentially important because of their adaptability for POF-based short span optical local distribution networks. The advantage of using polymeric materials is that they are easy to fabricate, low cost, and they enable us to carry out a variety of optical functions by attaching active elements to the polymer system. Several rare-earths have been successfully embedded into POFs and superfluorescence and amplified spontaneous emission (ASE) have been observed [2]–[4]. On the other hand, amplifiers with high gain and efficient lasers have been obtained with organic dye-doped POFs [5]–[9]. Since the discovery in 1996 of gain narrowing and stimulated emission in optically pump thin conjugated polymer films [10], the interest of these semiconducting polymers as gain medium has

been greatly increased due to their advantages, such as low-cost lasers, easy processability of materials and wide tuning range. Up to now, gain narrowing and lasing in conjugated polymers has been reported for many different polymer structures and device architectures, such as microcavities, slab waveguides, distributed Bragg reflectors, and distributed feedback structures [11]–[13]. Recently, it has been observed that POFs doped with semiconducting polymers can act as optical amplifiers [14], [15].The potential applications of these materials as amplifiers with the capacity of ultrafast optical switching, radiation sensors, and illuminators motivate the study of their optical properties [16], [17]. A detailed characterization of the optical properties of POFs doped with fluorene oligomers can be very useful to improve the performance of these devices. Although several studies on the spectroscopic properties of semiconducting polyfluorenes in different surrounding matrixes have been published [18]–[20], there are hardly any studies on their optical properties in fibers. It must be taken into account that in dye-doped fibers, in which light travels long distances, there are singular optical properties caused by self-absorption of the dopant molecules, stimulated emission, and nonlinear optical effects.

The aim of this study is to investigate the optical properties of fluorene oligomers embedded in plastic optical fibers by using steady-state spectroscopy. We have analyzed the dependence of the optical spectra with dopant oligofluorene, emission wavelength, excitation wavelength, direction of excitation (longitudinal or transverse), and light propagation distance through the fiber. From the analysis of the emission spectra as a function of the distance traveled by light, we have characterized the optical loss in these doped optical fibers. This method based on transverse excitation for measuring the optical attenuation in dye-doped fibers was first proposed in 1999 by Kruhlak and Kuzyk [21], [22], and it has been used to characterize the emission from a polymer optical fiber doped with a mixture of two different rhodamine dyes [23]. Since this method is nondestructive, it constitutes an alternative technique to the traditional methods like the cut-back method or the bulk measurement.

The paper is organized as follows. The experimental techniques are described in Section II. The spectroscopic properties of the doped fibers are analyzed and discussed in Section IIIA. and the dependence of the emission spectra on propagation distance is analyzed in Section IIIB. Finally, the summary of the paper is presented in Section IV.

II. EXPERIMENTAL DETAILS

In our experiments, we have investigated four fluorene oligomers of different lengths: a trimer (3), a pentamer (5),

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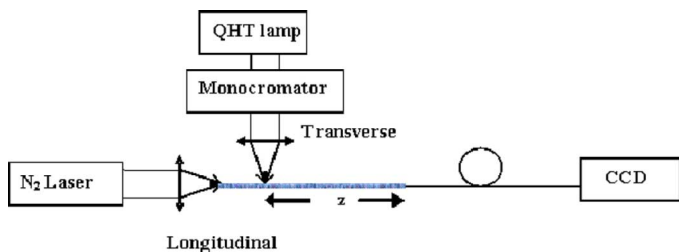Digital Object Identifier 10.1109/JLT.2008.2010274

Fig. 1. Experimental setup for the measurements of spectra with "longitudinal" and "transverse" excitation. The position of the sources is interchangeable.

Fig. 2. Absorption ($- \times -$) and emission (solid line) spectra of PFO-doped fiber. Note that the absorption and emission spectra are overlapped in the region 410–425 nm. The emission spectrum was obtained exciting the sample at 337 nm.

a heptamer (7), and a long chain (polymer PFO) of (9,9 dioctylfluorene), embedded in standard poly(methylmethacrylate) (PMMA) plastic optical fiber. The preparation of the doped POF is depicted elsewhere [14]. The concentration of the oligomers (Trimer, Pentamer, and Heptamer) in PMMA is about 0.02 wt% and the concentration of polymer PFO dopant is 0.003 wt%. The core diameter of the fibers is 970 μm, whereas the cladding thickness is 20 μm. Fibers were cut to 17–18 cm lengths, and the fiber ends were carefully polished by hand with polishing papers.

Fig. 1 illustrates the optical setup used to measure optical spectra (emission and excitation bands) of samples upon longitudinal or transverse excitation. The pumping light sources used were a pulsed Nitrogen laser (4 ns pulse width, 10 Hz repetition rate, and 470 μJ/pulse energy) and a 250 W Quartz Tungsten Halogen lamp in combination with a motorized monocromator (Cornerstone 260 1/4 m). This device allowed the excitation of the samples at wavelengths ranging from 300 to 600 nm. The impinging beam was focused onto the fiber by an objective lens (20$\times$/0.4 NA) and the output light from the fiber end was measured by a grating with 2048 charge-couple device (CCD) sensor array (AvaSpec-2048 Fiber optic spectrometer). We used a probe fiber of 250 μm of diameter to connect the doped fiber with the spectrometer. The transmitted emission spectra (upon transverse excitation) as a function of propagation distance through the doped fiber were measured by varying the incident point on the fiber. This was achieved by translating the doped fiber perpendicularly across the light source by means of an automatized long-range linear stage. All spectra have been corrected for the response of the detection system. The absorption spectra of the doped fiber were recorded on a Cary 50 UV–Vis spectrophotometer equipped with a fiber optic coupler accessory. In these measurements the lengths of the fibers were about 6 cm so that the absorption band of the dopant embedded in the fiber could be detected.

Atomic Force Microscopy (AFM) images were recorded at room temperature using a Nanoscope IIIa Multimode Scanning Probe Microscope (Veeco Instruments, Santa Barbara, CA). AFM was operated in tapping mode. Samples of plastic optical fiber were adhered to a sample mounting disk and tested using a phosphor-doped silicon cantilever (*Rtespa* model, Veeco) with nominal spring constant of 40 N/m and operating frequency of 300 kHz. The small radius of the probes ($R < 10$ nm) allowed the acquisition of high resolution phase images.
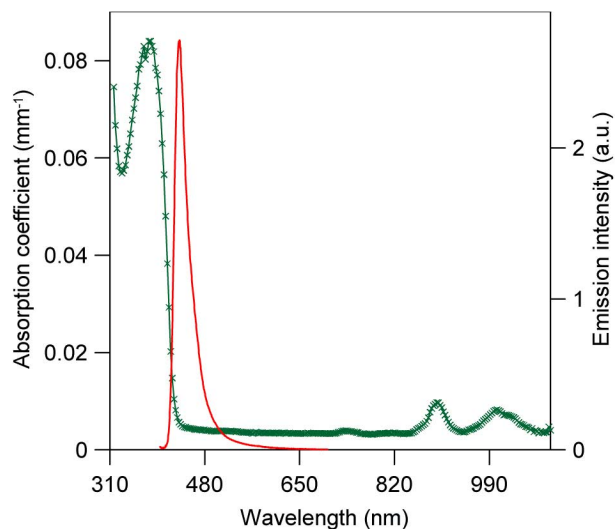
III. RESULTS AND DISCUSSION

A. Spectroscopic Analysis

The absorption spectra of the doped fibers are described by the characteristic absorption bands of the PMMA matrix in the near-infrared region together with an absorption band that peaks about 380 nm, which corresponds to the $S_0 \rightarrow S_1$ transition of the organic dopant embedded in the POFs. As an example, we show in Fig. 2 the absorption spectrum of the polymer PFO-doped fiber in the 300–1100 nm region. It can be observed that the band corresponding to the dopant is superimposed on the UV absorption edge of the PMMA. Due to the low concentration of the dopant, the obtained values for the absorption coefficient of this band in the PFO-doped fiber are lower compared to those obtained in the oligomers-doped fibers. Note that the lengths of measured fibers are very short (about 6 cm). Under these conditions, the power mode distribution of the fiber has not yet reached the equilibrium condition, and consequently, the attenuation obtained may be higher than that obtained by using long segments as demanded by the standard cut-back method. This fact must be taken into account particularly in order to assess or reproduce the absorption coefficients corresponding to the region where the dopant does not absorb [24].

The emission spectra of all fibers upon longitudinal or transverse excitation at 337 nm are presented in Fig. 3. The spectra are characterized by a broad and featureless band that corresponds to the $S_1 \rightarrow S_0$ transition. The spectra are markedly redshifted compared with those corresponding to the same active dopants embedded in thin films and in solutions [14]. The peak wavelengths and the full-width at half-maximum (FWHM) of the bands are displayed in Table I. The spectral separation between the absorption and emission (the Stokes shift) is small in all samples, but it is enough to provide low absorption in the
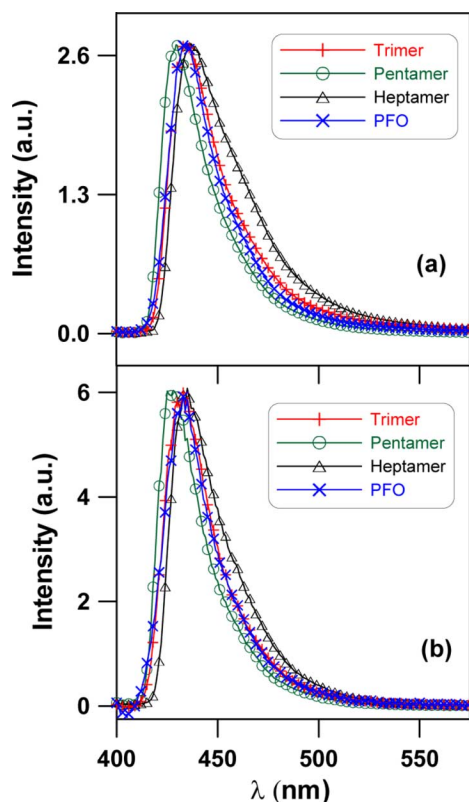
Fig. 3. Emission spectra of active polymer fibers doped with Trimer, Pentamer, Heptamer, and PFO: (a) upon longitudinal pumping, (b) upon transverse pumping. The corrected spectra have been normalized to have the same peak intensity.

TABLE I
PEAK WAVELENTHS AND FULL WIDTHS AT HALF MAXIMUM OF EMISSION SPECTRA

	Long λ_{peak} (nm)	Long $\Delta\lambda$ (nm)	Trans λ_{peak} (nm)	Trans $\Delta\lambda$ (nm)	ΔE_s (nm)
TRIMER	435	30	433	28	49
	(435)	(31.5)			
PENTAMER	430	27	428	26	48
	430	(27)			
HEPTAMER	437	38	435	30	47
	(438)	(42)			
PFO	435	28	433	26.5	55
	(435)	(29.5)			

Peak wavelengths (λ_{peak}) and full widths at half maximum ($\Delta\lambda$) of the emission spectra of Trimer, Pentamer, Heptamer and, PFO doped fibers upon (Long) longitudinal excitation and (Trans) transverse excitation at $\lambda_{ext}=337$ nm. The values in parenthesis correspond to λ_{peak} and $\Delta\lambda$ of the emission spectra obtained by exciting the fibers at $\lambda_{ext}=390$ nm. ΔE_s is the Stokes shift of the transition $S_0 \Leftrightarrow S_1$. It has been calculated from emission spectra upon longitudinal excitation.

spectral region of the maximum emission. The small values of Stokes shift indicate that the environment in the region around the dopant molecule is quite rigid. One can see that spectra recorded upon longitudinal excitation are shifted to red compared to the spectra of the same fiber recorded upon transverse excitation. This effect is observed in all fibers, and it is caused by the reabsorption and re-emission processes that take place in these materials due to the overlapping between the absorption and emission spectra (please refer back to Fig. 2). These processes will be analyzed afterward. It is worthwhile to notice that, when the excitation is transverse, the FWHM of the spectra decreases.

Taking into account that the characteristics of the optical spectra vary with the dopant concentration [7], we have only analyzed the emission properties of the fibers with the same oligomer concentration (0.02 wt% for Trimer, Pentamer, and Heptamer-doped fibers). Although the measured spectra are quite similar, the spectrum corresponding to the Heptamer-doped fiber is the most redshifted and the widest one. The higher width of the spectrum in this sample could be due to a higher inhomogeneous broadening. We have observed, in fact, inhomogeneity effects in these fibers by measuring both excitation spectra at different emission wavelengths and emission spectra at several pump wavelengths. Fig. 4 shows, as an example, the excitation spectra of the $S_0 \rightarrow S_1$ transition of the Trimer and Heptamer samples monitored at 460 and 500 nm (at the low-energy side of the emission band).

No remarkable changes have been detected by monitoring at wavelengths below 460 nm. The spectra monitored at 500 nm are redshifted as would correspond to the emission from the low-energy dopant molecules. Note that, apart from being redshifted, the spectrum corresponding to the Heptamer-doped fiber is narrowed. These spectral changes can be attributed to an inhomogeneous distribution of the organic molecules in the PMMA matrix.

The emission spectra obtained exciting at two different wavelengths are displayed in Fig. 5 for every fiber containing oligofluorenes. Whereas the emission spectra are independent of the excitation wavelength in the spectral region ($\lambda_{ext} < 400$ nm), both a redshift and a shoulder (at approximately 460 nm) are observed in the emission band by exciting at long wavelengths ($\lambda_{ext} = 400–410$ nm).

It can be observed that the changes in the emission spectra are slightly more pronounced as the molecular mass of the dopant molecules increases. A shoulder is clearly observed in the Pentamer and Heptamer-doped fibers whereas in the Trimer-doped fiber it is hardly detected. These results, together with a higher emission bandwidth observed in the Heptamer-doped fiber, suggest that the inhomogeneity in the samples increases with the dopant molecule length. Similar conclusions have been obtained from emission decay time measurements reported in [15]. In this case, an increase in the oligomer length leads to an increase of nonradiative deactivation rates, suggesting that the longer chain oligomers undergo some form of aggregation. In order to analyze the effect of the molecular mass of the dopant, AFM imaging of the fibers was carried out at room temperature. Fig. 6 shows the AFM images for undoped, Trimer, Pentamer, and Heptamer-doped samples. It can be observed a single homogeneous phase for undoped and Trimer-doped samples, indicating that fluorene oligomers of smaller size are miscible
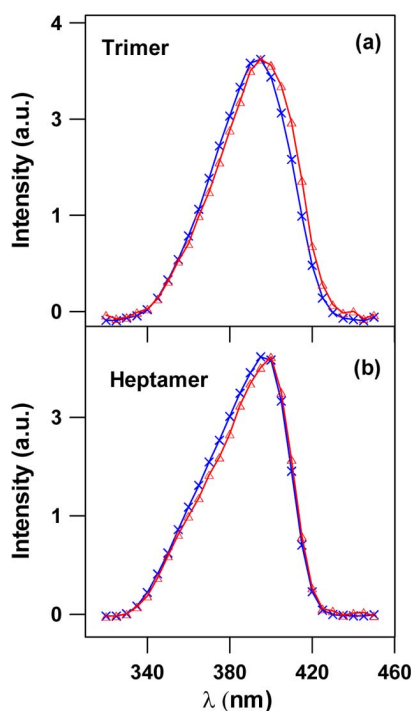
Fig. 4. Excitation spectra of the $S_0 \rightarrow S_1$ transition for (a) Trimer-doped sample and (b) Heptamer-doped sample. ($\times$) Spectra for luminescence monitored at 460 nm. ($\triangle$) Spectra for luminescence monitored at 500 nm (at the low-energy side of the emission band).

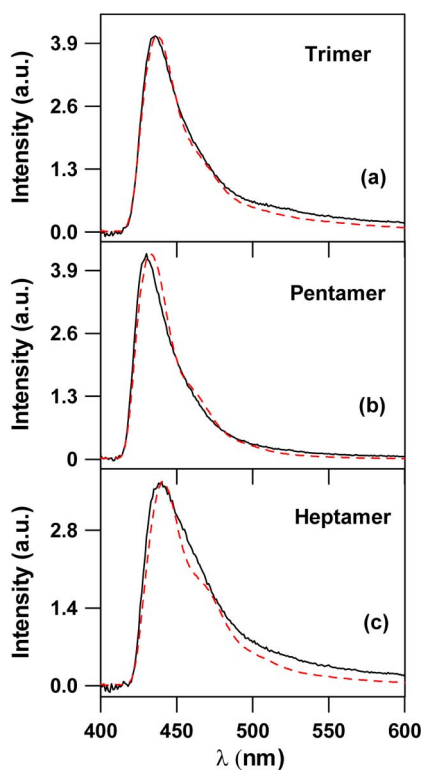
Fig. 5. (a) Emission spectra of Trimer-doped fiber by exciting at $\lambda_{ext} = 350$ nm (solid line) and at $\lambda_{ext} = 405$ nm (dashed line). (b) Emission spectra of Pentamer-doped fiber by exciting at $\lambda_{ext} = 350$ nm (solid line) and at $\lambda_{ext} = 405$ nm (dashed line). (c) Emission spectra of Heptamer doped fiber by exciting at $\lambda_{ext} = 350$ nm (solid line) and at $\lambda_{ext} = 410$ nm (dashed line). The corrected spectra have been normalized to have the same peak intensity.
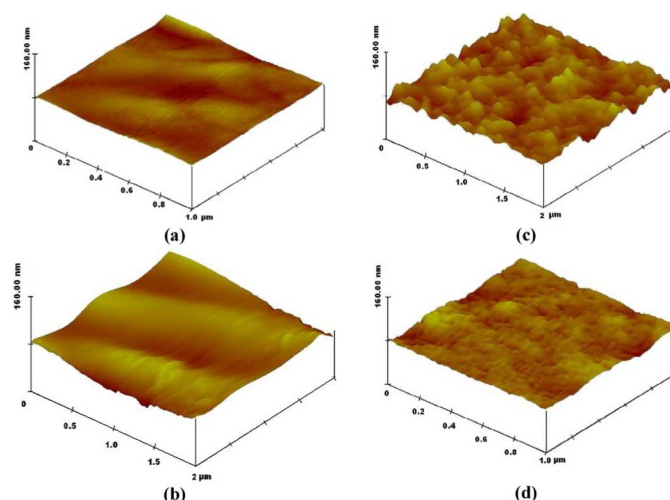

Fig. 6. AFM phase images (tapping mode) of PMMA optical fibers: (a) un-doped PMMA, (b) PMMA: Trimer 0.02 wt.%, (c) PMMA: Pentamer 0.02 wt.%, (d) PMMA: Heptamer 0.02 wt.%.

in the PMMA matrix. In contrast, the bright second phase detected in Pentamer and Heptamer-doped samples are indicative of nanophase separation. Phase separation leads to a distribution of organic molecules surrounded by a host matrix having a subtly different dielectric and chemical character. In consequence, the AFM results also reveal an enlargement of the inhomogeneity of the oligomer molecules in the PMMA matrix as the chain-length increases.

B. Dependence of Emission Spectra With Propagation Distance

The emission spectra upon transverse excitation at wavelengths $\lambda_{exc} = 337$ and 390 nm were recorded for various propagation distances from one edge of the fibers. Figs. 7 and 8 show the transmitted emission spectra measured as a function of propagation distance through the fibers doped with Trimer and Heptamer, respectively. It can be observed that as the propagation distance from one edge of the fibers increases, both the peak of the emission spectra is redshifted and the detected intensity at the end of the fibers decreases. The same behavior has been detected in other fibers.

Fig. 9 shows the variation of the first moment of the emission spectra as a function of the propagation distance through all the doped fibers. Due to the nonsymmetric shape of the emission spectra, the peak positions have been evaluated by the first moment of the spectra. As it can be seen, the peaks are redshifted as the propagation distance increases (about 60 mm). Except for the Heptamer sample, the shifts show a nonlinear behavior with the traveled path, with a soft tendency to saturation at longer distances. The redshift of the emitted signal has its origin in the overlapping of the absorption and emission spectra of the oligofluorenes. The spectral overlap region between the absorption spectrum and emission spectrum was illustrated in Fig. 2 for the PFO-doped sample. As the emission light is propagated through the doped fiber, the portion of emission spectrum that overlaps, the shorter wavelength part of the spectrum, is self-absorbed giving rise to a re-emission process in the longer wavelength part of the spectrum. These processes produce the red-
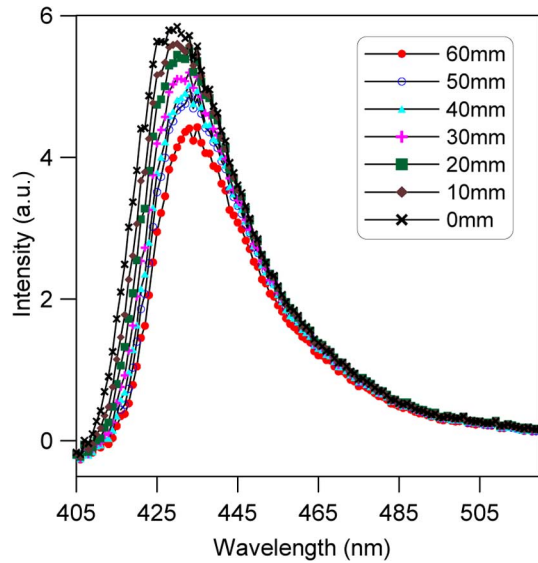
Fig. 7. Transmitted emission spectra as a function of the propagation distance through the Trimer-doped fiber. The point of the fiber closest to the detector has been normalized to zero. The excitation wavelength was $\lambda_{\text{ext}} = 390$ nm.

Fig. 9. Redshifted of the first moment of the emission spectra with propagation distance. (a) Trimer-doped fiber, (b) Pentamer-doped fiber, (c) Heptamer-oped fiber, (d) PFO-doped fiber. Emission spectra were obtained by exciting the samples at $\lambda_{\text{ext}} = 337$ nm. The point of the fiber closest to the detector were normalized to zero.
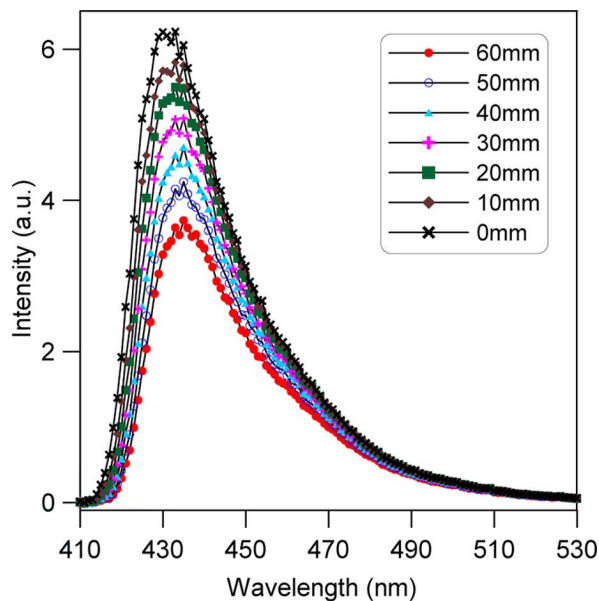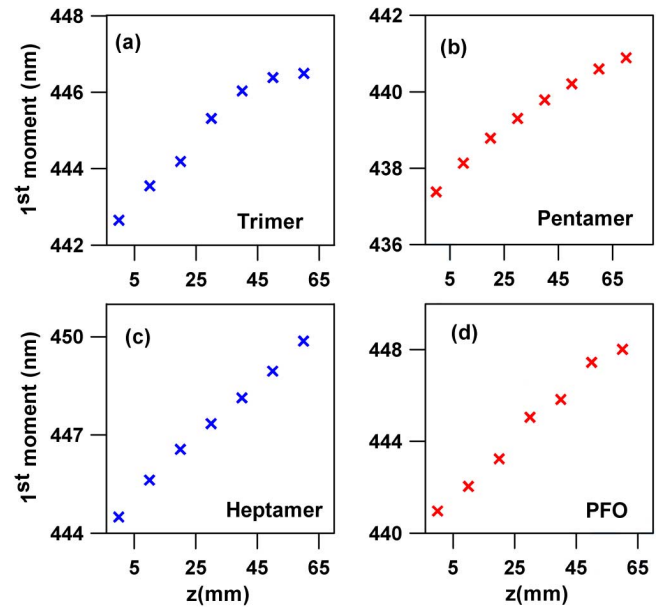

Fig. 8. Transmitted emission spectra as a function of the propagation distance through the Heptamer-doped fiber. The point of the fiber closest to the detector has been normalized to zero. The excitation wavelength was $\lambda_{\text{ext}} = 390$ nm.
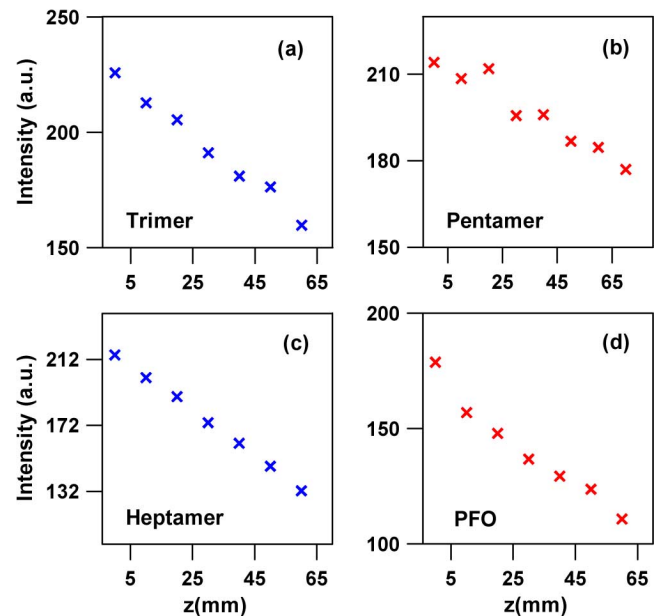

Fig. 10. Decrease of the spectrally integrated intensity with propagation distance. (a) Trimer-doped fiber, (b) Pentamer-doped fiber, (c) Heptamer-doped fiber, (d) PFO-doped fiber. Emission spectra were obtained by exciting the samples at $\lambda_{\text{ext}} = 390$ nm. The point of the fiber closest to the detector were normalized to zero.

shift of emission spectra. The diminishing of the spectrally integrated intensity detected in all samples is displayed in Fig. 10 and it is caused by the loss mechanics such as absorption and scattering processes.

By assuming the illuminated portion of the fiber a homogeneous light source, that is, a plane-wave source, the light intensity at wavelength λ after propagating z-distance from the source, $I_s(\lambda)$, is given by

$$I(z, \lambda) = I_s(\lambda) \exp[-\alpha(\lambda)z] \qquad (1)$$

where $\alpha(\lambda)$ is the linear attenuation coefficient at that wavelength. This expression describes approximately how the light is propagating through the fibers giving the upper limit on $\alpha(\lambda)$. The lower limit on $\alpha(\lambda)$ would be described by taking a point-source model. In this case, the light intensity after propagating z-distance would be given by an expression slightly different
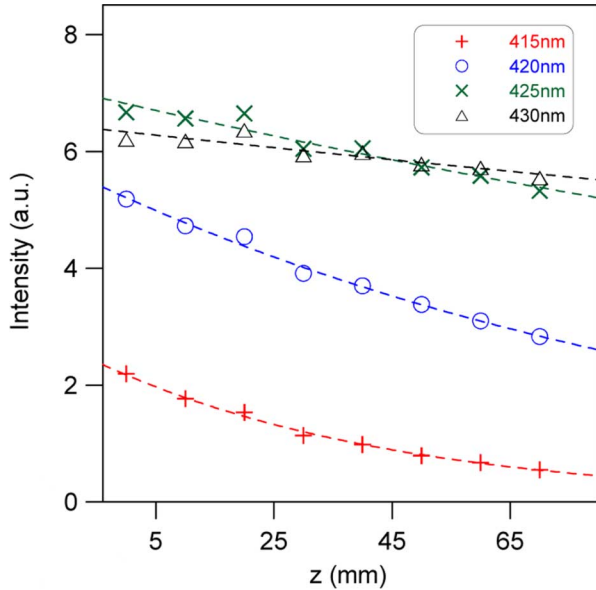
Fig. 11. Transmitted intensity as a function of the propagation distance through the Pentamer-doped fiber at different emission wavelengths. The dashed lines are the fittings to (1). The coefficients of determination (R^2) of the fittings are $(+)\lambda_{\mathrm{em}} = 415\,\mathrm{nm}$, $R^2 = 0.9965$, $(o)\lambda_{\mathrm{em}} = 420\,\mathrm{nm}$, $R^2 = 0.9933$, $(x)\lambda_{\mathrm{em}} = 425\,\mathrm{nm}$ $R^2 = 0.9389$, $(\triangle)\lambda_{\mathrm{em}} = 430\,\mathrm{nm}$ $R^2 = 0.8353$. The excitation wavelength was $\lambda_{\mathrm{ext}} = 390\,\mathrm{nm}$.

Fig. 12. Linear attenuation coefficient $\alpha(\lambda)$ determined from (1) for all doped fibers studied in the range $410\,\mathrm{nm} < \lambda < 440\,\mathrm{nm}$. Lines are the absorption coefficients calculated by means of a Cary 50 spectrophotometer. Solid line corresponds to Pentamer-doped fiber and dashed line to Heptamer-doped fiber.

from (1) [21]. Values estimated for $\alpha(\lambda)$ in dye-doped polymeric fibers from a plane-wave source or from a point source differ by less than 10% [22].

We display in Fig. 11 the transmitted emission intensity versus the propagation distance at several emission wavelengths for the Pentamer-doped fiber. It can be demonstrated that the curves corresponding to the low wavelength part of the emission spectrum, where there is absorption–emission overlap (412–422 nm), present an exponential behavior with a good fitting to (1). However, as we move away from that region toward longer wavelengths, the variation of the intensity with the distance is softened [with poor fittings of the curves to (1)] becoming almost constant at longer wavelengths. The nonexponential behavior of the curves at the region of nonoverlapping in the emission spectrum is caused by the re-emission of light from the shorter wavelength part of the spectrum to that region. Consequently, the re-absorption and re-emission phenomena provide lower attenuation for longer wavelengths at the larger distances. An exponential behavior of these curves implies that the attenuation coefficient is constant along the length of the fiber, whereas a nonexponential one suggests that the coefficient is dependent on the propagated distance through the fiber. From the fitting to (1) of the curves with a clear exponential decay in the region of overlapping of the emission spectrum, we have calculated the linear attenuation coefficients $\alpha(\lambda)$ for that spectral region. The results obtained for all the doped fibers can be seen in Fig. 12. It can be noticed that the attenuation of the Pentamer-doped fiber is lower than that of the other fibers. This effect is only observed in the displayed spectral region (the tail of the absorption bands) and it is due to the blue-shift of the spectrum of the Pentamer sample compared to the spectra
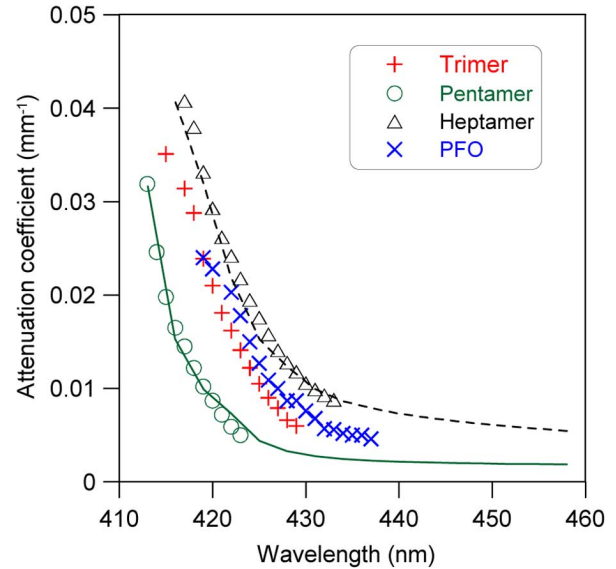
of the other fibers. The values for $\alpha(\lambda)$ obtained from (1) agree very well with those calculated by using a spectrophotometer. This fact indicates that the transverse-excitation emission measurement method (also called side-illumination fluorescence measurement technique [22]) gives attenuation values similar to those obtained by using short segments of fibers, that is, attenuation values corresponding to fibers that have not reached the equilibrium mode distribution. However, these attenuation values are more useful to analyze doped POFs since most of the measurements of active fibers are performed on short lengths. All these results validate the side-illumination fluorescence technique as a useful method to measure the optical attenuation in doped fibers.

IV. SUMMARY

The optical properties of four fluorene oligomers embedded in POFs have been characterized by using steady-state spectroscopy. From the dependence of the optical spectra with excitation and emission wavelengths, we conclude that there is an inhomogeneous distribution of the oligofluorene molecules in the POFs, which is enlarged as the chain length of the dopant molecule increases. The variation of the emission spectra with the traveled distance through the fibers has been measured and analyzed. As the propagation distance increases, there is a redshift of the emission spectra in addition to a decrease of the spectrally integrated intensity. From the analysis of these data, we have characterized the losses in the doped fibers. It has been found that in the longer wavelength side of the emission spectrum, there is a decrease of attenuation at larger distances of propagation in the fibers. These results can be useful in order to obtain an appropriate design of a plastic optical amplifier or laser based on fluorene oligomers-doped POFs.

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