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ORIGINAL ARTICLE

Photophysical Properties of some 1,3,4-Oxadiazole Derivatives Containing Phenolphtalein, Fluorene and Bisphenol A Units

Mihaela Homocianu[1] · **Alina M. Ipate**[1] · **Corneliu Hamciuc**[1] · **Anton Airinei**[1]

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Abstract

The photophysical properties of the three 1,3,4-oxadiazole derivatives containing fluorene (Ox-FL); fluorene and phenolphtaleine (Ox-FL-FF); or fluorene and bisphenol A (Ox-FL-BPA) moieties in the main chain were investigated by the fluorescence and absorption spectroscopy in different solvents and in the solid state. The electronic absorption spectra included a strong absorption band located in the 270–395 nm region, with a maxima around at 302 nm. The fluorescence excitation spectra were also characterized by one broad band, appearing in the wavelength range of 220–340 nm. All samples displayed the emission bands around 356–373 nm and exhibit high quantum yields ranged from 31.61 to 90.77%, in chloroform solution. The sensitivity of the emission spectra on medium characteristics (polarity, acidity and basicity) were evaluated by using the Catalan solvent scale and the fluorescence titration with a dilute acid solution.

Keywords Emission quantum yields · Light-emitting materials · Optoelectronic applications · Photophysical properties

Introduction

Oxadiazole based molecular assemblies are an interesting and continuously developing area of research. These compounds demonstrate interesting luminescent properties, emitting blue to green light with high quantum efficiency depending on the substituents attached to the oxadiazole ring. Given facile synthesis and the possibility of appending different π-conjugated groups new oxadiazole derivatives are intensively investigated for biomedical applications [1–3] and in polymer field [4–11] due to their advanced optoelectronic properties. It is known that the introduction of electron-transporting 1,3,4-oxadiazole units (Ox) into the polymer improves their optical properties.

Phenolphthalein (FF) is an organic compound that is widely used in food products and as a laxative in various pharmaceutical products [12]. Also, the aromatic and bulky structure of FF unit conferee good mechanical and thermal properties of the final products which contain this unit.

Generally, the compounds containing fluorene groups (FL) are materials used in numerous photo-sensitive applications, like as solar cells [13, 14] and photovoltaic applications [15, 16]. The presence of bisphenol A (BPA) in the many consumer products (toys, drinking containers, medical equipment and electronics, etc.) [17], in the natural environment [18, 19], as well as their biological activities (such as antimicrobial, anti-inflammatory, antibacterial, analgesic and antibacterial potential etc.) [20] justified the investigation of the photophysical behavior of different derivatives containing bisphenol A [6].

By combination of varying chromophore units it would be possible to provide a good high performance properties for new created systems in comparison of to the each individual component. Some of main features of the obtained materials containing in the structural chain, 1,3,4-oxadiazole ring together with other voluminous moieties like phenolphthalein (FF), fluorene (FL) and bisphenol A (BPA), includes improved solubility, good processability into thin, flexible films and coatings etc. These aspects and the above mentioned specific features of the FF, FL and BPA structural units motivated authors to investigate the photophysics of the dilute solutions of systems containing these units.

The aim of this paper was thus to investigate how structural features and the environmental factors (medium characteristics) influence the photo-physical behavior of these materials.

✉ Mihaela Homocianu
michalupu@yahoo.co.uk

[1] "Petru Poni" Institute of Macromolecular Chemistry, 41A, Grigore Ghica Voda Alley, 700487 Iasi, Romania

Figure 1 presents the chemical structures of the investigated copolymers in this study.

Experimental

Materials and Methods

A series of aromatic copolyethers containing 1,3,4-oxadiazole rings and fluorene groups was prepared by nucleophilic substitution polymerization technique of 9,9-bis(4-hydroxyphenyl)fluorene or of different amounts of this compound and an aromatic bisphenol, such as 4,40-isopropylidenediphenol or phenolphthalein, with 2,5-bis(fluorophenyl)-1,3,4-oxadiazole. These syntheses were carried out at elevated temperature in N-methyl-2-pyrrolidone (NMP) solution, in the presence of anhydrous potassium carbonate, according to a previously published method [21–23]. Its molecular structure and purity were established from different spectroscopic studies, like 1H NMR and FTIR [21–23]. All copolymers showed good solubility in conventional organic solvents, such as N-methyl-2-pyrrolidone, N,N-dimethylacetamide, chloroform, tetrahydrofuran and 1,2-dichloroethane [21].

The steady state ultraviolet visible absorption spectra were measured on a Schimadzu 3600 spectrometer in: tetrahydrofuran (THF, ε=7.58), chloroform ($CHCl_3$, ε=4.81), 1,2-dichloroethane (CH_2Cl_2, ε=10.42), N-methyl-2-pyrrolidone (NMP, ε=33.0), N,N-dimethylacetamide (DMAc, ε=37.8), N,N-dimethylformamide (DMF, ε=38.25) and dimethyl sulfoxide (DMSO, ε=46.7) solutions. The steady state emission and excitation spectra were measured using Perkin Elmer LS55 fluorescence spectrometer. The fluorescence quantum yields (QY%) measurements, were performed on an FLS 980 (Edinburgh Instruments, Ltd.) spectrofluorimeter equipped with a Quanta-phi integrating sphere accessory and at λ_{exc} corresponding to the maximum of the first absorption band, using dilute solutions with absorbance lower than 0.1. Both the sample and reference were excited at the same wavelength. All solvents were spectroscopic grade. Films of polymers were prepared by casting a solution of 5% concentration of polymer from chloroform solutions on to glass plates, followed by drying at room temperature for 24 h under a Petri dish. The resulting flexible films were stripped off the plates by immersion in hot water for 2 h. The excitation wavelength used for the fluorescence measurements was absorption maxima of the compounds in respective solvents. Protonation was studied by fluorescence spectroscopy of the polymers dissolved in tetrahydrofuran solution by titration with dilute solution of 1 N HCl. All the measurements were performed at room temperature, in a quartz cuvette, with 1 cm optical path length.

Results and Discussion

Absorption and Fluorescence Spectra

The UV-vis absorption and fluorescence spectra of the three new 1,3,4-oxadiazole derivatives containing: fluorene (Ox-FL); fluorene and phenolphtaleine (Ox-FL-FF); or fluorene and bisphenol A (Ox-FL-BPA) moieties in the main chain were measured. These spectra were recorded in selected solvent solutions and in the thin solid films. The obtained spectral data are listed in Table 1.

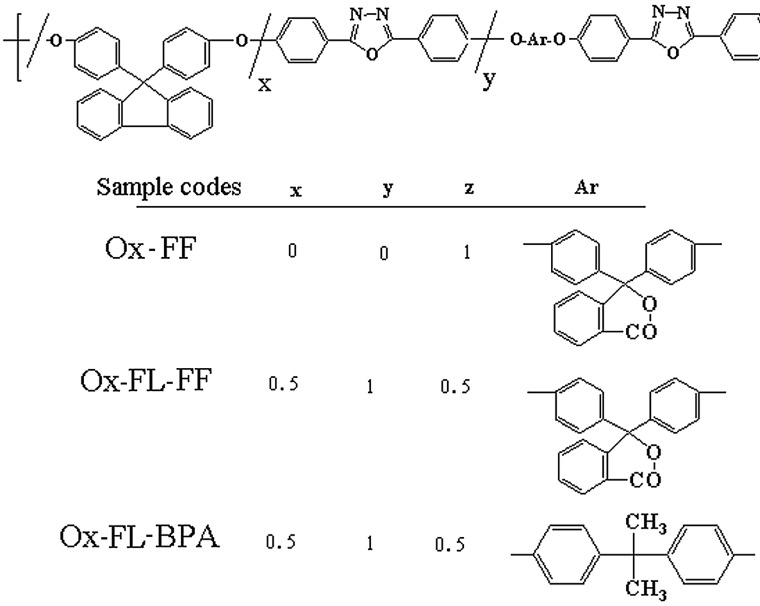

Fig. 1 Chemical structure of the polymers Ox-FF, Ox-FL-FF and Ox-FL-BPA

Table 1 Optical data of all polymers in different solvents

Code	Media	λ_{max}^{abs}, nm	λ_{max}^{ex}, nm	λ_{max}^{em}, nm	Stokes shifts, eV	QY, %
Ox-FF	THF	300	287sh;293;306	356.5;372	0.65	91.29
	$CHCl_3$	303	287sh;293;306	363;374	0.67	90.77
	CH_2Cl_2	301	287sh;293;307	360;373	0.67	76.58
	NMP	303	290sh;307	361;375	0.65	18.08
	DMAc	302	288sh;306.5	360;373	0.66	97.28
	DMF	302	285sh;294sh;306	361;373	0.67	62.25
	DMSO	303	285sh;294sh;306	363;374.5	0.67	52.92
Ox-FL-FF	THF	301	287sh;293;306	357.5; 372.5	0.65	–
	$CHCl_3$	306	287sh;293;306	359;374.5	0.59	79.47
	CH_2Cl_2	302	287sh;293;307	359;373	0.65	–
	NMP	309	290sh;307	361;373	0.57	–
	DMAc	299	288sh;295;306.5	361;374	0.71	–
	DMF	301	286sh;293; 306.5	361;373	0.68	–
	DMSO	304	287sh;293;306	363; 373.5	0.66	–
Ox-FL-BPA	THF	302	287sh;293;306	357;372.5	0.63	–
	$CHCl_3$	309	287sh;293;306	360;374	0.56	31.61
	CH_2Cl_2	306	287sh;293;306.5	360;373.5	0.60	–
	NMP	309	288sh;295;308	362.5; 373.5	0.59	–
	DMAc	301	288sh;295;307	362;373	0.69	–
	DMF	303	287sh;293;306.5	362;373	0.66	–
	DMSO	299	290sh;308	364.5;372	0.74	–

sh shoulder

Figure 2 displays the absorption spectra of the Ox-FL-FF sample in different solvents. These spectra exhibited a highly intense absorption band in the range of 270–395 nm, with a maximum centered around 303 nm, which can be attributed to the π-π* electronic transition of the conjugated polymer backbones (see Table 1). Minor shifts (variations of λ_{max}^{abs} are in the range of 6 nm in $CHCl_3$ solution, see Table 1) of the absorption maxima of the derivatives containing the most voluminous - fluorene units (Ox-FL-BPA and Ox-FL-FF samples, see Fig. 2a) were observed. The Ox-FL-BPA polymer in chloroform solution has the most red-shifted absorption maxima among the three copolymers, due to extended conjugation of copolymer backbone including voluminous units. Instead, when changing the solvent polarity the variations of λ_{max}^{abs} were found up to 10 nm. Even if the shifts induced by effects of medium characteristics (solvents) are rather small, the solvents influence strongly other spectral parameters such as: shape of the absorption and emission spectra, full width at half maximum (FWHM), Stokes shift, the intensity of absorption or emission bands. The full width at half maximum ranged between 4754 and 7799 cm-1 in the absorption spectra for Ox-FL-FF sample. The value of the full width at half maximum (FWHM) corresponds to the width of the band at the intensity equal to $0.5A_{max}$ and was calculated from the distance between the curve points at the peak half maximum level according to following relation, $\Delta\lambda_{FWHM} = \lambda_2\left(\frac{A_{max}}{2}\right) - \lambda_1\left(\frac{A_{max}}{2}\right)$, where Amax is the value of the maximum absorbance; $\lambda_2\left(\frac{A_{max}}{2}\right)$, $\lambda_1\left(\frac{A_{max}}{2}\right)$ are the wavelengths to which is half maximum absorbance. The absorption spectral widths for Ox-FL-FF sample in selected media decreased in the follow order: $\mathrm{FWHM}_{abs}^{CH_2Cl_2}$ (6441 cm^{-1}) < $\mathrm{FWHM}_{abs}^{DMSO}$ (7501 cm^{-1}) < $\mathrm{FWHM}_{abs}^{DMF}$ (7308 cm^{-1}) < $\mathrm{FWHM}_{abs}^{THF}$ (7211 cm^{-1}) < $\mathrm{FWHM}_{abs}^{CHCL_3}$ (6776 cm^{-1}) < $\mathrm{FWHM}_{abs}^{NMP}$ (4754 cm^{-1}) < $\mathrm{FWHM}_{abs}^{DMAc}$ (7799 cm^{-1}) (see Fig. 2).

Figure 3a and b show the fluorescence excitation spectra of the Ox-FL-FF sample in DMAc solution and in various solvents. Compared with the peaks of the absorption spectra (~ 302 nm), the shape of fluorescence excitation spectra were quite similar (they overlap with the absorption spectra), but the positions of the maxima for the band in the fluorescence excitation spectra was toward to longer wavelengths by 6 nm (from ~ 302 to 306 nm in DMAc solution, see Table 1). This observation indicates that the nuclear configurations of the ground and excited states of these compounds were identical. Also, this behavior suggests that the emission originates from the studied compounds and not from any species that could appear in the system. The values of the excitation and emission maxima of the investigated compounds were summarized in Table 1.

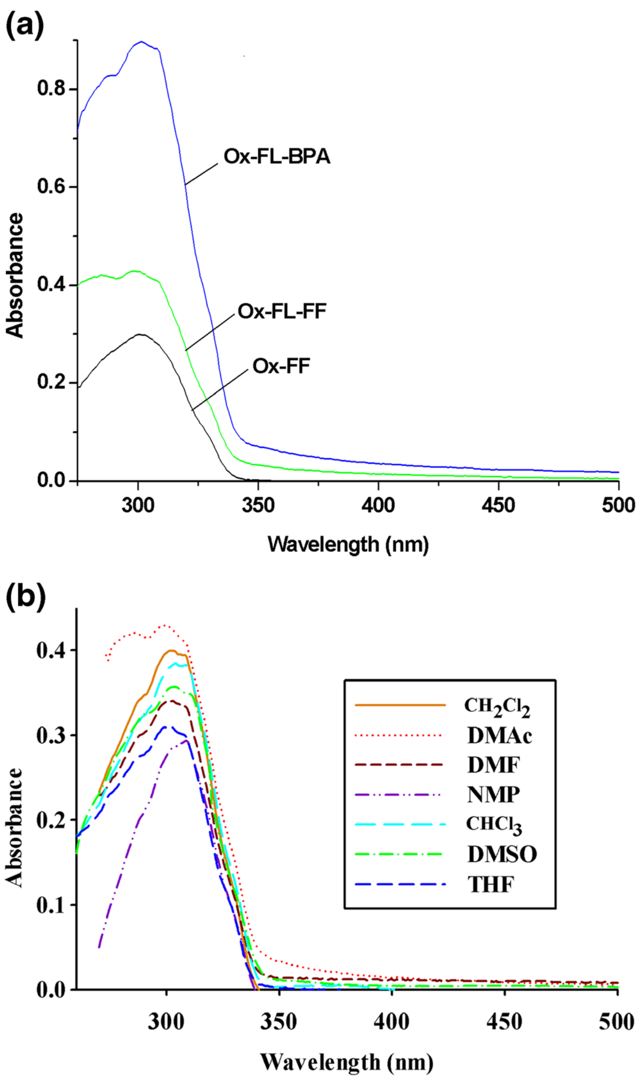

Fig. 2 **a** The absorption spectra of all samples in DMAc solution and **b** the absorption spectra of the Ox-FL-FF sample in various solvents

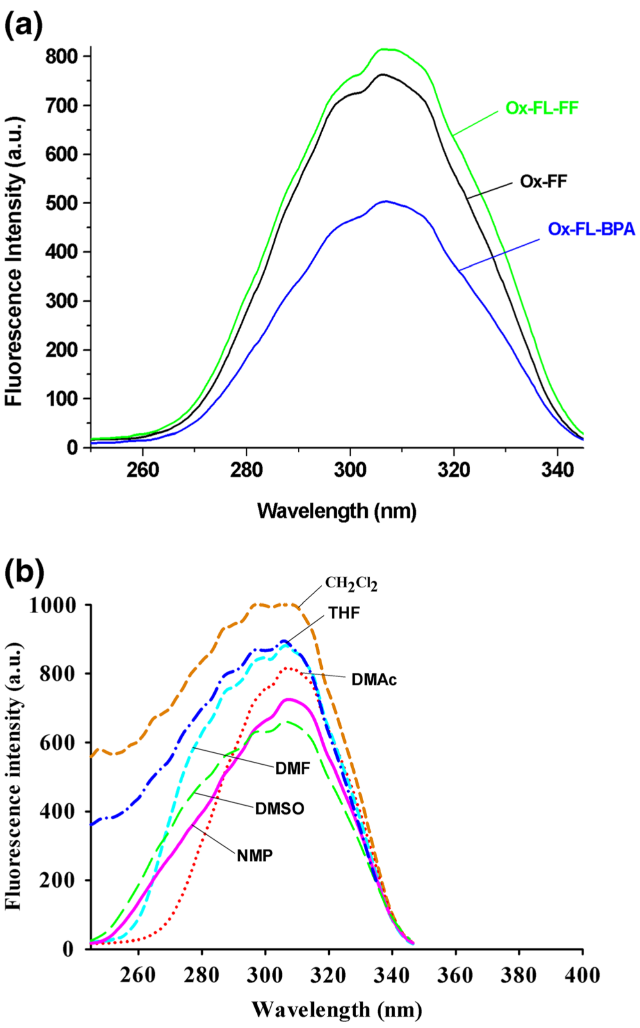

Fig. 3 **a** The fluorescence excitation spectra of the of all samples in DMAc solution and **b** the fluorescence excitation spectra Ox-FL-FF polymer in selected solvents

The fluorescence emission spectra of all investigated copolymers were measured in different solutions with increasing solvent polarity. Upon excitation at the corresponding wavelength of the absorption maxima, the obtained emission profile of all copolymers consists in a broad emission band with two main discernible maxima at around 360 and 373 nm, and a poorly resolved shoulder (around 330 nm), as can be seen from Fig. 4. Although, CH_2Cl_2 and $CHCl_3$ are chlorinated solvents, the doublets in the emission spectra from CH_2Cl_2 solution are well-resolved compared to that in chloroform, due to different values of the solubility parameter and to specific interactions [24]. The fluorescence emission bands of Ox-FL-BPA sample with BPA in the molecular structure were slightly red-shifted in comparison with those of the Ox-FF and Ox-FL-FF copolymers, that can be attributed to the specific solute-solvent interactions and indicating relatively longer conjugation in this system. Instead, when changing the solvent polarity, only minor shifts of the emission maximum of all copolymers were observed (10 nm).

The emission spectra of the investigated compounds were also measured in a solid state (thin films, as shown in Fig. 5). The emission bands obtained in spectra of the films were slightly broader than the corresponding of the spectra from the solution and slightly red shifted of the emission bands occurs due to intermolecular interactions (conformational changes of the polymer backbones from solution to the solid state or just increased π-π-stacking) in the solid state. The emission maxima of polymers' films ranged from 378 to 392 nm (see Fig. 5). They were around 3 to 19 nm red-shifted relative in comparison with those in the in solution (Table 1). These indicate that these polymers have a slight tendency to form aggregates.

The fluorescence emission spectra were recorded under different excitation wavelengths. As shown in Fig. 6, upon excitation with different excitation wavelengths (from 303 to

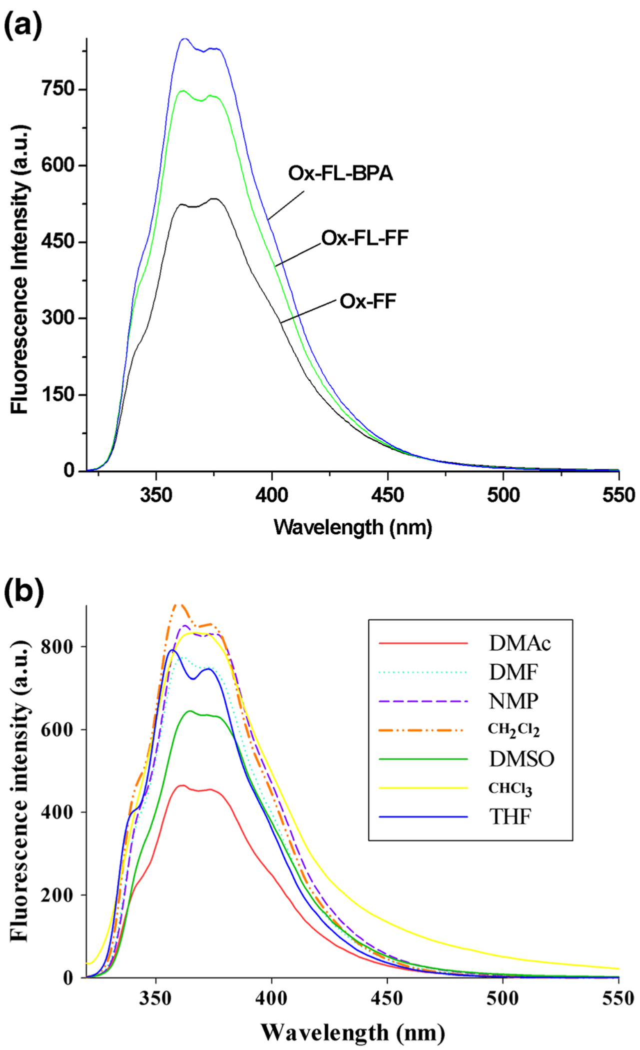

Fig. 4 The fluorescence emission spectra of the Ox-FL-BPA sample in various solvents with different polarities

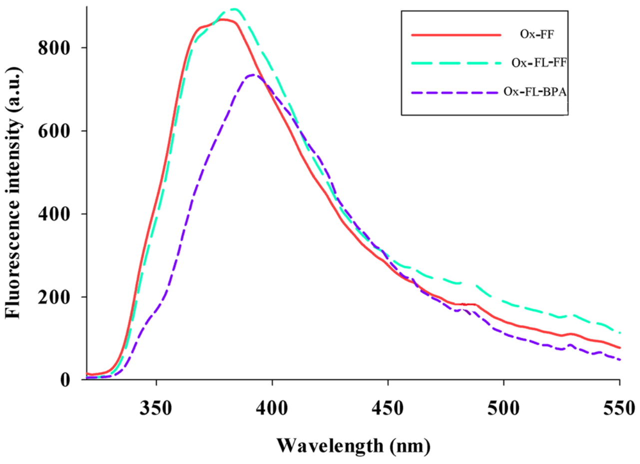

Fig. 5 The fluorescence emission spectra of Ox-FF, Ox-FL-FF and Ox-FL-BPA thin films

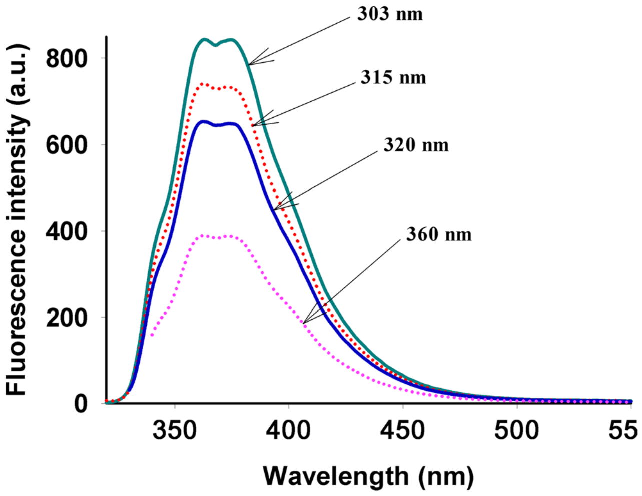

Fig. 6 The fluorescence emission spectra of Ox-FF sample in DMSO solution, with an increase of excitation wavelegth from 303 to 360 nm

330 nm), the intensity of the emission band of Ox-FF sample in DMSO solution decreases, without shifting. Similar behavior was observed in all investigated samples and indicates that the fluorescence emission wavelength of these compounds does not depend on the excitation wavelength.

Other interesting characteristics for potential applications of investigated materials are the Stokes shifts (SS, eV) and fluorescence quantum yields (QY, %). The Stokes shift values were estimated from the difference in energy between the band maxima of the absorption and the lower-energy emission and they were ranging between about 0.63 and 0.74 eV according to the structures of the polymers and the medium characteristics (Table 1).

The composition of the copolymers leads to significant differences (self-quenching due to intermolecular electronic interactions) in the values of the fluorescence quantum yields (ranged from 31.61 to 90.77%) in chloroform solution. Thus, QY has higher values of Ox-FF sample as compared to values for Ox-FL-FF and Ox-FL-BPA (see Table 1) compounds. Moreover, the values of QY were affected by medium characteristics (solvents), as exemplified for Ox-FF sample in seven solvents with different polarities in Table 1.

Solvatochromism

In order to analyze the effects of the medium characteristics on the photophysical properties of these samples, we used the Catalan multi-term solvent scale [25], that allowing distinction and evaluation of the individual intermolecular interactions of a given system. The Catalan solvent scale is an experimental solvent scale that takes into account polarizability (SP), dipolarity (SdP), acidity (SA) and basicity (SB) of the medium and allows to obtain specific separation of solvent effects. For the determination of the solvent parameters (SA, SB, SP and SdP) from the Catalan scales, the same proper probe/

Table 2 The Catalan solvent parameters taken from reference [25]

Solvents	SA	SB	SP	SdP
THF	0	0.591	0.714	0.634
$CHCl_3$	0.047	0.071	0.783	0.614
CH_2Cl_2	0.03	0.126	0.771	0.742
NMP	0.024	0.613	0.812	0.959
DMAc	0.028	0.65	0.763	0.987
DMF	0.031	0.613	0.759	0.977
DMSO	0.072	0.647	0.83	1

homomorph pair was used and thus pure effects were evaluated. The solvent effects on the physicochemical observable Y property, evaluated using the Catalan solvent scale, can be expressed by Eq. (1):

$$Y = Y_0 + aSA + bSB + cSP + dSdP \tag{1}$$

where: SA, SB, SP and SdP, describe the acidity, the basicity, the polarizability and the dipolarity, of solvents, respectively. Y_0 is the property of the investigated molecules in the gas phase. The values of the regression coefficients *a, b, c* and *d* reflect the influence of the solvent descriptors (SA, SB, SP and SdP) on the Y spectral properties of the copolymers. The SA, SB, SP and SdP parameters for each solvent were listed in Table 2 and were taken from reference [22].

The obtained regression coefficients {Y_0, a, b, c and d} and the multilinear correlation coefficient, R^2, for all copolymers were presented in Table 3. The calculations were performed with Origin software. The results indicate a strong influence of solvent acidity (hydrogen-bond donating ability) and polarizability on the emission properties of copolymers, that was reflected in the large values of the a and c coefficients. The negative sign of *a* coefficient for all samples indicates a positive solvatochromism. With increasing polarity of the solvents a bathochromic shift of the emission maxima was observed.

The qualitative agreement between the calculated (with regression coefficients obtaining fitting Eq. (1)) and experimental values for the emission maxima of sample Ox-FL-FF was related by $R^2 = 0.998$ (Fig. 7).

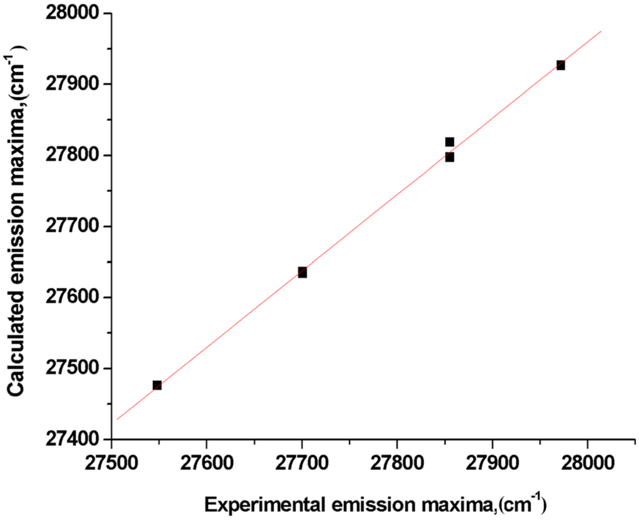

Fig. 7 Linear relationship ($R^2 = 0.998$) between the experimental and calculated emission maxima of Ox-FL-FF sample obtained by a multiple linear regression analysis according to Eq. (1)

Sensitivity to Acidity of the Medium

Quantitatively, the dominant effect (obtained from Catalan solvatochromic analysis) of acidity of the medium on the solvatochromic behavior of investigated compounds, was analyzed by fluorimetric titration of the solutions of these samples with a dilute solution of aqueous hydrochloric acid (HCl). Figure 8 displays the emitting spectra of the Ox-FF sample in THF solution, before and after addition of different amounts of a dilute solution of HCl. It is observed that the spectra are essentially identical in shape (the maximum of the fluorescence band remains at the same value), but the fluorescence intensity decreases with an increase in acid concentration, this indicates a protonation of the nitrogen atoms from 1,3,4-oxadiazole rings. When 1100 μL HCl was added the emission intensity was almost completely quenched.

Table 3 The regression coefficients obtained from the correlation between the emissions spectral data of the studied samples and the Catalan parameters of the solvents

Code	Y_0, (cm^{-1})	*a*, (cm^{-1})	*b*, (cm^{-1})	*c*, (cm^{-1})	*d*, (cm^{-1})	R^2
Ox-FF	28,942.05	−4179.44	236.44	−1263.53	−243.53	0.884
Ox-FL-FF	28,690.43	−2639.14	−81.52	−530.97	−462.29	0.999
Ox-FL-BPA	29,373.52	−2367.38	47.53	−1327.15	−706.45	0.996

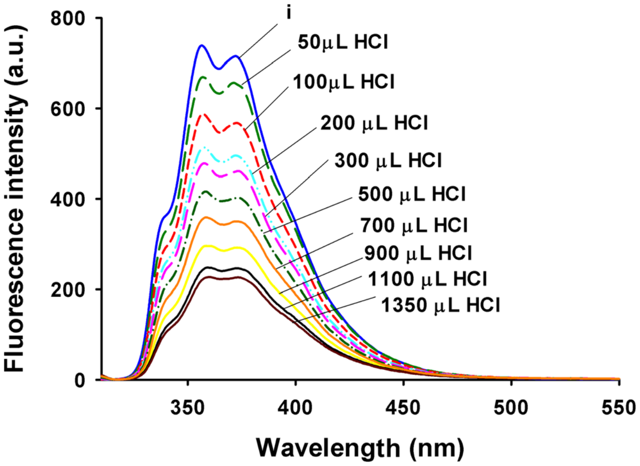

Fig. 8 The fluorescence emission spectra of the Ox-FF sample in THF solution, recorded without and after were added the different amounts of HCl

Conclusions

The exploration of photophysics of molecular assemblies consisting of particular sequences of organic chromophores present a real interesting in the field of real-world of applications. In this paper, we investigated the photophysical properties of three highly fluorescent copolymers containing 1,3,4-oxadiazole, phenolphtalein, fluorene and bisphenol A moieties in the main chain (Ox-FL, Ox-FL-FF and Ox-FL-BPA) and were correlated with their structure. These compounds displayed emission bands around 356–373 nm and have high quantum emission yield values in different solvents. The wavelengths of the maxima of the absorption and emission spectra of these compounds show only modest shifts with the solvent polarity, as expected for typical π-π* transitions. However, a red shift of the absorption with the increase in the conjugation of the samples is observed in chloroform solutions. Multilinear regression analysis based on Catalan multi parameter solvent scale indicates that these compounds were very sensitive to the acidity of the microenvironment (to the pH of the solution). The sensitivity of these materials to acidity was investigated by fluorimetric titration of copolymer with a dilute solution of HCl.

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