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Specific spectral characteristics of some phenylquinoxaline derivatives



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1. Introduction

Poly(aryl-ether)s represent a class of polymers well known as engineering thermoplastics with desirable properties, such as melt and solution processability, chemical stability and high glass transition temperature. The aromatic part of these polymers contributes to thermal stability and good mechanical properties, and the ether linkages facilitate the polymer processing while maintaining oxidative and thermal stability [1]. The introduction of heterocyclic units, such as phenylquinoxaline or 1,3,4-oxadiazole rings, into the main chains of aromatic poly(aryl-ether)s leads to polymers with good solubility, increased glass transition temperature and high thermal stability [2,3]. Aromatic poly(aryl-ether)s containing these heterocyclic units show improved solution and melt processing characteristics as compared to their counterparts containing only aromatic rings. These polymers can be prepared by nucleophilic displacement to form ether linkages during the polymerization reaction. For example, poly(aryl-ether phenylquinoxaline)s can be prepared by polymerization of bisphenols with bis(fluorophenylquinoxalines) [3], by reacting self-polymerizable phenylquinoxaline monomers [4,5], or by the reaction of bisphenols containing phenylquinoxaline rings with aromatic electrophiles such as 4,4'-difluorobenzophenone or 2,5-bis(4-fluorophenyl)-1,3,4oxadiazole [6].

Aromatic poly(oxadiazole ether)s are of special interest due to their various physico-chemical properties and their potential use as electrontransporting materials or electroluminescence materials in organic light emitting diodes. Due to their optoelectronic properties, high thermal

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ABSTRACT

Absorption and emission spectral characteristics of a bis(hydroxyphenylquinoxaline) Q, and two aromatic polyethers containing phenylquinoxaline rings, Q-DBF and Ox-Q, were studied in various solvents. Q, Q-DBF and Ox-Q display two absorption bands, while a poly(1,3,4-oxadiazole-ether) Ox-BisA, exhibits a single absorption band. Their fluorescence spectra $(2.5 \times 10^{-6} \text{ mol L}^{-1})$ were characterized by an emission band between 380 and 520 nm. At slightly lower concentrations $(6.4 \times 10^{-7} \text{ mol L}^{-1})$ and under excitation with light corresponding to the highest energy absorption band, Q-DBF and Ox-Q in ethyl acetate and cyclohexane solutions exhibited simultaneously dual emission bands in the near-UV (\approx 355 nm) and blue region (\approx 432 nm). The emission characteristics were quantified by CIE indices (in x, y color coordinates) for the investigated systems. Values of fluorescence quantum yields ranging from 15.93 to 49.83% in chloroform media were obtained.

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stability and good mechanical properties, poly(oxadiazole ether)s play an important role in materials science [7–9].

Generally, dual fluorescence of different compounds is of great significance in their application in multifunctional sensor microarrays and other microscale sensor systems [10]. The dual fluorescence of many chromophores can be attributed to different processes occurring in the system, such as tautomerism [11], formation of two different excited states [12–15], conformational changes, excimer/exciplex formation [16], excited-state intramolecular proton transfer (ESIPT) [17], and twisted intramolecular charge transfer (TICT) [18].

The present work focuses on the characterization of a series of phenylquinoxaline derivatives, one of them containing 1,3,4-oxadiazole rings, by using optical absorption, fluorescence, emission quantum yields and emission lifetime measurements. The photophysical properties of the phenylquinoxaline derivatives have been investigated in dilute solutions of various solvents (cyclohexane, toluene, chloroform and ethyl acetate) and in solid state. Their emission properties in chloroform were compared using a chromaticity diagram.

2. Experimental

2.1. Materials

Bis(hydroxyphenylquinoxaline), Q, was synthesized by the reaction of 4-hydroxybenzil with an aromatic tetraamine, 3,3'-diaminobenzidine, according to a previously published method [19]. 2,5-Bis(4fluorophenyl)-1,3,4-oxadiazole was prepared from 4-fluorobenzoic acid and hydrazine hydrate, in polyphosphoric acid, according to the literature [20].

Poly(phenylquinoxaline ether ketone), Q-DBF, and poly(phenylquinoxaline-1,3,4-oxadiazole ether), Ox-Q, were synthesized by the conventional aromatic nucleophilic substitution polymerization technique of bis(hydroxyphenylquinoxaline) with 4,4'-difluorodiphenyl ketone or with 2,5-bis(4-fluorophenyl)-1,3,4-oxadiazole, respectively. Poly(1,3,4-oxadiazole ether), Ox-BisA was prepared by the same synthetic procedure from 2,5-bis(4-fluorophenyl)-1,3,4-oxadiazole and 2,2-bis(4-hydroxyphenyl)propane [21].

Q-DBF Yield: 90%. FTIR (film, cm⁻¹): 3060 (C–H aromatic), 1653 (C = 0), 1592 (C–C aromatic), 1344 and 1310 (phenylquinoxaline ring), 1236 (aromatic ether group). ¹H NMR (400 MHz, CDCl₃, δ): 8.60 (2H, s) 8.31 (2H, d), 8.26 (2H, d), 7.81 (4H, d), 7.55 (8H, m), 7.37 (6H, d), 7.05 (8H, m).

Ox-Q Yield: 92%; FTIR (film, cm⁻¹): 3057 (C–H aromatic), 1598 (C–C aromatic), 1342 (phenylquinoxaline ring), 1241 (aromatic ether group), 1012 and 977 (oxadiazole ring). ¹H NMR (400 MHz, CDCl₃, δ): 8.61 (2H, s), 8.32 (2H, d), 8.25 (2H, d), 8.11 (4H, d), 7.61 (8H, d), 7.42 (6H, s), 7.16 (4H, d), 7.07 (4H, d).

Ox-BisA Yield: 87%. FTIR (film, cm^{-1}): 3060 (C–H aromatic), 2980 and 2880 (isopropylidene group), 1600 (C–C aromatic), 1240 (aromatic ether group), 1020 and 980 (oxadiazole ring). ¹H NMR (400 MHz, CDCl₃, δ): 8.06 (2H, d), 7.27 (2H, d), 7.10 (2H, d), 7.00 (2H, d), 1.55 (6H, s).

2.2. Methods

The inherent viscosities (η_{inh}) of the polymers were determined with an Ubbelohde viscometer, by using polymer solutions in N-methyl-2-pyrrolidone (NMP), at a concentration of 0.5 g dL⁻¹, at 20 °C.

The ¹H NMR spectra were recorded on a Bruker DRX 400 spectrometer, equipped with a 5 mm multinuclear inverse detection probe, operating at 400 MHz. ¹H chemical shifts are reported in δ units (ppm) relative to the residual peak of the solvent (ref. ¹H, CDCl₃: 7.26 ppm).

The molecular weight was determined by gel permeation chromatography (GPC) using UV and SLS detectors and PLgel 5micro Mixed C Agilent and PLgel 5micro Mixed D Agilent Columns. Measurements were carried out with polymer solutions in chloroform using chloroform as eluent. Polystyrene standards of known molecular weight were used for calibration.

Fourier transform infrared (FTIR) spectra were recorded on an FTIR Bruker Vertex 70 spectrometer at frequencies ranging from 400 to 4000 cm⁻¹. UV–Vis absorption spectra were obtained with a Shimadzu 3600 spectrophotometer. Steady state fluorescence emission and excitation spectra were measured with a Perkin Elmer LS55 spectrofluorimeter. Fluorescence emission and excitation spectra were recorded by exciting at the corresponding wavelengths of the absorption and emission maxima, respectively. All the solvents used were of spectrophotometric grade. Thin films for photophysical studies were prepared by casting chloroform solutions of the polymers onto glass substrates. Fluorescence lifetime and emission quantum yields were measured using an FLS 980 Edinburg Instruments spectrofluorimeter. Fluorescence lifetime measurements were carried out by the standard timecorrelated single-photon counting method. The excitation was provided by an Eddinburgh Picosecond Pulsed Diode Laser (EPL-375), at 375 nm. The criteria for a good fit for lifetimes were estimated by statistical parameters namely: χ^2 values close to unity and residual trace which was symmetrical about the zero axes. Fluorescence quantum yields were measured using an integrating sphere. The absorbance of the solutions was maintained around 0.1. All measurements were made at room temperature, in a quartz cuvette, with 1 cm optical path length.

3. Results and discussion

3.1. Synthesis

The polycondensation reactions were carried out at high temperature in NMP, in the presence of anhydrous potassium carbonate. The nucleophilic aromatic substitution of an aryl halide with a phenoxide is the most common route to high performance, high temperature poly(aryl



Ox-BisA

Fig. 1. Chemical structure of bis(hydroxyphenylquinoxaline), Q and polymers Q-DBF, Ox-Q and Ox-BisA.

ether)s. It has been demonstrated that heterocycles, as well as the ketone group, can activate aryl halides towards nucleophilic aromatic substitution polymerization generating high molecular weight poly(arylether)s. The oxadiazole moiety and the ketone group in 2,5-bis(4fluorophenyl)-1,3,4-oxadiazole or 4,4'-difluorodiphenyl ketone, respectively, act as activating groups: they can accept a negative charge and lower the activating energy for the displacement of the *p*-substituted fluoro group through a Meisenheimer complex [6,21,22]. The structures of monomer Q and of polymers Q-DBF, Ox-Q and Ox-BisA are illustrated in Fig. 1.

The inherent viscosity values of Ox-BisA, Ox-Q and Q-DBF, determined in NMP, were of 0.39, 0.54 and 0.48 dL g^{-1} , respectively. The values of weight-average molecular weight (Mw) ranged between 8800 and 21,000 g mol⁻¹, the number-average molecular weight (Mn) values between 16,800 and 66,000 g mol⁻¹ and the polydispersity Mw/Mn between 1.91 and 3.14 (Table 1).

The polymers were soluble in polar solvents, such as NMP, *N*,*N*-dimethylacetamide, and *N*,*N*-dimethylformamide, and in chlorinated solvents, such as chloroform or dichloroethane. They were insoluble in acetone or even dimethyl sulfoxide. Polymer Q-DBF was partially soluble in DMF (Table 2). The good solubility of these polymers was due to the presence of flexible ether linkages which determined an increase in macromolecular chain flexibility. Also, in the case of polymers Ox-Q and Q-DBF, the bulky phenylquinoxaline units disturbed the packing of the macromolecular chains, thus facilitating the diffusion of small molecules of solvent which led to a better solubility.

3.2. UV-visible spectral characterization

The optical properties of bis(hydroxyphenylquinoxaline), Q, and polymers Q-DBF, Ox-Q and Ox-BisA, were investigated by UV–Vis absorption and photoluminescence measurements. The electronic absorption spectra of phenylquinoxaline compounds in chloroform solution are shown in Fig. 2a. Bis(hydroxyphenylquinoxaline), Q, and polymers Q-DBF and Ox-Q displayed two main absorption bands an intense band (Band I) in the wavelength range 250–325 nm and the second less intense band (Band II) located around 325–425 nm (Fig. 2a). The higher energy bands (Band I) are assigned to the absorption of phenyl rings from the phenylquinoxaline unit, diphenyl ketone group (from Q-DBF) or diphenyl-1,3,4-oxadiazole unit (from Ox-Q sample), while the low-energy bands (Band II) are due to the π - π * transition of the phenylquinoxaline moiety [23].

The spectral shape and band positions of the compound Q are different from those of its derivatives, Q-DBF and Ox-Q, in chloroform solution. The absorption characteristics of these derivatives present the strongest differences in the region of Band I. It is noted that this absorption band exhibits a prominent maximum (centered on 275 nm for Q, for example) accompanied by a shoulder on both the left and right sides of the spectrum (Fig. 2a). The I- and II-absorption bands were fitted using the deconvolution of the initially overlapped absorption spectrum (Fig. 2b). The spectral differences (position of the shoulders) can be the consequence of possible intermolecular interactions within the systems and the electron delocalization. The longest wavelength absorption band of Ox-Q and Q-DBF (Band II) is blue-shifted relative in relation to that of Q. As expected, a hypsochromic shift in the absorption maximum of Q-DBF occurred, as opposed to that of Ox-Q, due to a

Table 1	
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Inherent	viscosity	and	GPC of	lata	of the	polymers.
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Polymer	η_{inh} (dLg ⁻¹)	Mn (g mol ⁻¹)	Mw (g mol ⁻¹)	Mw/Mn
Q-DBF	0.48	19,700	56,000	2.84
Ox-Q	0.54	21,000	66,000	3.14
Ox-BisA	0.39	8800	16,800	1.91

 $\eta_{inh} =$ inherent viscosity (20 °C, 0.5 g/100 mL, NMP).

Table 2	
Solubility	of polymers

Joiubility	01	polymers.	

Polymer	Solven	t						
	NMP	DMAc	DMF	Chloroform	DCE	Acetone	DMSO	
Q-DBF	+	+	+-	+	+	_	_	
Ox-Q	+	+	+	+	+	_	_	
Ox-BisA	+-	+	+	+	+	-	_	

 $\label{eq:NMP} NMP = N-methyl-2-pyrrolidone; DMAc = N,N-dimethylacetamide; DMF = N,N-dimethyl-formamide; DCE = dichloroethane; DMSO = dimethyl sulfoxide; + soluble; - insoluble; +- partial soluble.$

higher degree of conjugation provided by the oxadiazole unit compared to that imparted by the diphenyl ketone group. In contrast with the spectral pattern of phenylquinoxaline derivatives, Ox-BisA presents only an absorption band around 300 nm (Table 3) due to the oxadiazole chromophore, confirming that the second absorption band originates from the phenylquinoxaline core. Some photophysical characteristics of the investigated compounds in various solvents are listed in Table 3.

3.3. Steady-state emission spectra and fluorescence lifetime decays

The emission spectra of compounds Q, Q-DBF and Ox-Q in chloroform solution are presented in Fig. 3. As may be noted from Fig. 3 and Table 3, it can observe that these compounds show an emission maximum ranging from 410 to 432 nm, depending on the solvent. The fluorescence intensity of these bands decreased when the samples were excited with the wavelength corresponding to the absorption maximum of Band II, as compared with the emission intensity obtained under excitation with the wavelength of Band I. As in the case of the absorption spectra, the emission maxima of Q-DBF and Ox-Q are blueshifted in comparison with that of Q, and the fluorescence maximum of Q-DBF is also blue-shifted, as opposed to that of Ox-Q (Fig. 3). The



Fig. 2. (a) Absorption spectra of Q, Q-DBF and Ox-Q in CHCl₃ solution. (b) Gaussian decomposition of the absorption spectra of Q-DBF, in chloroform solution.

 Table 3

 Spectral data and Stokes shifts of studied compounds in various solvents.

Code	Solvent	λ_{abs} , nm		$\lambda_{em}^{\#}$, nm	Stokes	
		Band I	Band II		shifts, cm ⁻¹	
Q	CHX	275	390	_ ^a	-	
	Toluene	-	390	424, 445(sh)	2056.12	
	CHCl ₃	275	390	443	3067.66	
	EtAc	271	390	434	2599.55	
Q-DBF	CHX	275	364	_a	-	
	Toluene	-	370	412	2755.18	
	CHCl ₃	260, 292(sh)	370	417	3046.21	
	EtAc	264, 282(sh)	365	415	3300.87	
Ox-Q	CHX	-	_b	_b	-	
	toluene	-	384	422	2344.98	
	CHCl ₃	278(sh), 303	387	425	2310.38	
	EtAc	275	384	432	2893.51	
Ox-BisA	CHX	298	-	355	5388.03	
	toluene	-	383	338(sh), 357,	-	
				370, 422(sh)		
	CHCl ₃	307	-	340(sh), 360,	4795.51	
				374, 398(sh)		
	EtAc	300	-	335(sh), 355, 370	5164.31	

CHX-cyclohexane; CHCl₃-chloroform; EtAc-ethyl acetate; # – excitation with wavelength corresponding to the absorption maximum of Band II, at 2.5×10^{-6} mol L⁻¹ concentration; sh – shoulder peak.

^a No fluorescence emission was exhibited under excitation with wavelength corresponding to the absorption maximum of Band II.

^b Sample Ox-Q is insoluble.

Sample Ox-Q is module.

shape of the emission spectra of sample Q and its derivatives (Q-DBF and Ox-Q) in all studied solvents is similar. On the other hand, the band positions and intensities of the emission spectra strongly depend on the solvent nature. In the emission spectral data of the Q-DBF and Ox-Q compounds (Table 3), shifts to longer wavelengths of the emission bands are observed in toluene (412 nm, 422 nm), CHCl₃ (417 nm, 425 nm) and EtAc (415 nm, 432 nm). The emission maximum of Ox-Q (with diphenyl-1,3,4-oxadiazole) is more red-shifted as compared to that of sample Q-DBF (which contains a ketone unit) in the solvents used. The emissions of compounds Q, Q-DBF and Ox-Q in cyclohexane are practically very small under the excitation was done with the wavelength corresponding to the phenylquinoxaline ring.

The excitation spectra of the polymers under study were measured at fixed emission wavelengths (as listed in Table 3). Fig. 4 presents exemplified excitation spectra for Q, Ox-Q and Ox-BisA in toluene and cyclohexane solutions, respectively. For all samples, the shapes of the absorption and excitation spectra are similar and the maxima appear at around the same wavelengths.



Fig. 3. Emission spectra of Q (-, $1.22 \times 10^{-5} \text{ mol } L^{-1}$), Ox-Q (-, $2.5 \times 10^{-6} \text{ mol } L^{-1}$) and Q-DBF (•••, $6.03 \times 10^{-6} \text{ mol } L^{-1}$), in CHCl₃ solution.



Fig. 4. Excitation spectra for Ox-BisA in CHX and Q, Ox-Q in toluene solutions.

In the case of polymer Ox-BisA, the absorption spectrum consists of one band (inset in Fig. 5, Table 3), while the emission spectra (Fig. 5) present one band with two pronounced peaks, accompanied by two shoulders in CHCl₃, ethyl acetate, and toluene (Table 3), except the cyclohexane (where only one emission band is exhibited). The fluorescence intensity of Ox-BisA in CHCl₃ solution gradually decreased when the excitation light wavelength values increased (Fig. 5). It is obvious that the shape and band position of the emission spectra did not change with the excitation wavelength, indicating the presence of only one type of emitter in the conjugated chains [24].

At low concentration $(1 \times 10^{-7} \text{ mol } \text{L}^{-1})$ and under excitation with the wavelength corresponding to the absorption maximum of Band I, in ethyl acetate and cyclohexane, the two emission bands of derivatives Q-DBF and Ox-Q were observed. In order to understand the presence of dual emissions, the effect of excitation wavelength and concentration values on the emission spectra was investigated. Fig. 6 depicts the emission spectra for Ox-Q (in EtAc) and Q-DBF (in CHX), at 6.4×10^{-7} and 1.27×10^{-6} mol L⁻¹ respectively, under different excitation wavelengths. At the excitation wavelength of Band II (from the absorption spectra), Q-DBF in CHX displayed a single emission band situated at 409 nm (Fig. 6a). When Q-DBF in CHX was excited with λ_{ex} = 275 nm (Band I), dual emission bands were observed, one centered at $\lambda_{em} = 330$ nm and another band situated at 408 nm, with an I_{330}/I_{408} ratio of 0.336 (Fig. 6a). The first emission band around 330 nm is due to the locally excited (LE) state and the longer wavelength emission band arises from internal charge transfer (ICT). The fluorescence intensities of these bands depend on the excitation wavelength, indicating



Fig. 5. Emission and absorption spectra (inset plot) of Ox-BisA, in CHCl₃ solution, at different excitation wavelengths.



Fig. 6. Emission spectra of Q-DBF ($6.4 \times 10^{-7} \text{ mol } L^{-1}$) in CHX (a) and Ox-Q in EtAc ($1.27 \times 10^{-6} \text{ mol } L^{-1}$) (b), under different excitation wavelengths.

that these bands represent the emitting levels resulting from two different electronic states [25]. At excitation wavelengths shorter than 275 nm (260 and 250 nm), the intensity of the emission band increases, while the intensity of the second emission band decreases insignificantly (Fig. 6a). In the case of sample Ox-Q in EtAc solution, the effect of excitation wavelength values on the fluorescence intensity of the emission band is less pronounced (Fig. 6b). The emission maximum observed around 330 nm is assigned to the locally excited state [26]. The Stokes







Fig. 8. Fluorescence decay profile and residuals for compound Q in $CHCl_3$ solution, $\lambda_{ex} = 294$ nm; IRF = instrument response function.

shift values, for all samples in different solvents have been observed to vary in the range 2056–5388 $\rm cm^{-1}$ (Table 3) and to increase with increasing solvent polarity.

The emission spectra of the Q-DBF and Ox-Q derivatives in solid state (thin films) are shown in Fig. 7. The maxima associated to the emission band position ($\lambda_{em} = 446$ for Ox-Q and $\lambda_{em} = 425$ nm for Q-DBF) are red shifted by 10–21 nm, as compared to their corresponding dilute solution spectra, as a consequence of reduced rotational freedom of the polymer chains and a considerable intermolecular interactions that occurred in the solid state.

The emission decay curves of the investigated compounds give the information about lifetimes of the excited states of these systems. Fig. 8 illustrates the emission decay curves for Q in CHCl₃ solution. The results are consistent with single exponential fluorescence decay in each case. Luminescence lifetime values and chromaticity CIE coordinates of the Q, Q-DBF, Ox-Q and Ox-BisA compounds in chloroform solutions are listed in Table 4. Our results for fluorescence lifetimes, τ , (Table 4) are comparable with those of the other quinoxaline derivatives, e.g. for 2,3-bis(4-ethoxyphenyl)quinoxaline (EOQ) [27].

Photoluminescence quantum yields (QY) were measured in CHCl₃ solution for all the studied compounds and were determined to range between 15.93 and 49.83%, (Table 4). The quantum yield values were strongly affected by the presence of oxadiazole or biphenyl ketone units in the macromolecular chain. The CIE – Commission Internationale de l'Eclairage, (x,y) coordinates [28] of the studied derivatives in chloroform were determined based on the corresponding emission spectra, in order to compare the emission characteristics (Fig. 9). The chromaticity coordinates of phenylquinoxaline derivatives

Table 4	
Fluorescence lifetime and chromaticity coordinates of phenylquinoxaline derivatives	s in
CHCl ₃ solution.	

Sample	τ (ns)	χ^2	CIEx	CIEy	QY (%)
Q Q-DBF Ox-Q	0.94 0.50 0.60	1.096 1.033 0.998	0.150 0.161 0.165	0.083 0.052 0.074	15.93 49.83 22.36
Ox-BisA	-	-	0.163	0.016	-



Fig. 9. CIE chromaticity diagram, calculated from the emission spectra of Q (1), Ox-Q (2), Q-DBF (3) and Ox-BisA (4) samples in chloroform solution. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

in chloroform are situated in the blue region indicating that these polymers emit blue light (Table 4). The analysis of the CIE (x,y) coordinates indicates that the presence of oxadiazole and ketone units in the polymers shifts the emission bands of the samples towards shorter wavelengths in the following order: Q (λ_{em} –424 nm) > Ox-Q (λ_{em} –422 nm) > Q-DBF (λ_{em} –412 nm). The CIE index for the polymer solutions under study (Table 3) is close to the "blue" emission line.

4. Conclusion

The spectral characteristics of some phenylquinoxaline derivatives were investigated by determining the UV–visible absorption, emission and excitation spectra, fluorescence lifetimes and emission quantum yields. At low concentration and under excitation with light corresponding to the absorption maximum of Band I, the phenylquinoxaline derivatives show dual emission bands with more intense ICT band over LE emission. In solvents with different polarities, these bands show spectral shifts. The intensity of the observed dual emission bands changed with the excitation wavelength. The CIE chromaticity coordinates evaluated from the emission spectra for all these compounds confirmed the blue-emitting characteristic of these materials. The strong blue fluorescence of the compounds under study was influenced by the presence of diphenyl-1,3,4-oxadiazole or diphenyl ketone units and by the solvent nature.

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