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View Article Online DOI: 10.1039/C6RA10467C

CROYAL SOCIETY OF CHEMISTRY

Journal Name

ARTICLE

Received 00th January 20xx, Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

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Perylene Diimide Derivatives as Red and Deep Red-Emitters for Fully Solution Processable OLEDs

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We report on the photophysical characterization of two solution-processable red-emissive perylene diimide molecules and on their use in fully solution assembled OLEDs. The two emitters contain sterically hindered aromatic naphthalene or acenaphtene substituents on the perylene core. These fused rings limits the perylene diimide intermolecular π - π interactions in the solid state due to highly sterically hindered effects, while preserving an extended conjugation between the substituents and the perylene core. Indeed, these features generate a broad absorption for both perylene derivatives along with an efficient red and deep red emission in the solid state for the PDIs with naphthalene and acenaphtene, respectively. The performances of both emitters were tested on OLEDs, where the active layer is a film of bulk PDI, fabricated by simple solution processing. The devices with acenaphtene-substituted perylene diimide provide deep red electroluminescence with emission wavelength at 690 nm, CIE coordinates of (x=0.69, y=0.29) and show the best efficiency reported so far for OLED based on PDI fluorescent emitters.

Introduction

Organic light-emitting diodes (OLEDs) have gained increased attention due to their vast applications in future electronic technologies [1]. Although OLED displays are already present on the market in smart devices and TVs, the production process implies a high-cost vacuum deposition of the functional materials. Therefore, the focus of the current research is on lowering the OLEDs manufacturing costs and developing new solution processable emitting materials. In terms of fabrication, it is well established that the solution processing, such as spin coating or ink-jet printing [2], would guarantee a more reliable, cost-effective route in which also the wastage of the materials is substantially reduced. Recently, Lee and co-workers demonstrated that using the same materials, solution processed devices can provide enhanced performances over vacuum-deposited ones [3]. Regarding the materials, in this technology, both polymers [4] and molecular structures [5] have been exploited. While conjugated polymers offer high luminescence and solubility, they can suffer from issues associated with high polydispersity, batch-to-batch variations and impurities that are difficult to remove. Conjugated organic small molecules or oligomers offer great advantages like well-defined molecular structure, production reproducibility and easy purification by standard techniques.

Among the three primary red, green and blue emitters [6], fluorescent red materials still remain behind in terms of efficiency

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and purity. Most of red/deep red OLEDs are based on phosphorescent organometallic complexes[7], but their application in long term mass production is restricted by poor processability, resource-limited metals and high costs, and therefore, it is crucial to develop metal-free organic red emitting materials. Up to now, the organic red emitters are generally dopants from the dicyanomethylene series [8], polyacene and chromene-based materials [9] while the non-doped pure molecular red emitters are rather rare [10-12]. The majority of these molecules contains π extended polycyclic aromatic hydrocarbons, susceptible to aggregation by π - π stacking, making them difficult to dissolve in common solvents, which leads to formation of extended crystalline domains in the solid state. These domains can lead to luminescence quenching and act as carrier traps, which can raise the operational voltage of a device [13]. This is also the case of perylene diimides (PDIs), an important class of pigments widely used in fiber and automotive industry. Besides their industrial application, PDIs also combine a good absorption in the visible region with high fluorescence quantum yields in solution, together with excellent thermal and photochemical stability. Because of their rigid planar backbones, they are typically characterized by strong π - π stacking, depending on the chemical structure, which reduces both their solubility and their solid state photoluminescence efficiency [14]. Although these stacking features are fundamental for n-channel materials in organic field effect transistors [15], in order to extend their application in optoelectronic devices such as organic solar cells [16], fluorescent sensors [17], solar concentrators [18] and organic light emitting diodes [19], it is mandatory to limit and prevent the PDI Aggregation-Caused Quenching (ACQ) [20]. Despite the high demand for red emissive materials for OLEDs, only few examples of devices whose active layer is based on pervlene diimides have been so far reported [19,21,22]. Pan et al. constructed simple devices

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Electronic Supplementary Information (ESI) available.See DOI: 10.1039/x0xx00000x

DOI: 10.1039/C6RA10467C Journal Name

ARTICLE

consisting of emitting layer built solely with dendronized perylene derivatives. Although the best OLED surpassed 0.1% of external quantum efficiency and had red emission centered at 650 nm, the synthetic pathway is rather complicated [23]. The majority of these works relate on PDIs dispersed in polymeric or molecular matrices and show device performances decreasing by increasing the PDIs concentration, due to ACQ phenomena [19]. PDI-host films often suffer from unbalanced charge transport properties, due to the good electron transporting ability of PDIs, requiring the use of charge and exciton blocking layers in OLED architectures [21].

We have recently reported on the functionalization of PDIs core with bulky substituents, leading to molecules with a limited selforganization tendency and weak intramolecular interactions via π - π stacking [24b]. The substitution in bay positions with sterically hindered substituents induces a distortion of the perylene core and impedes the formation of π - π interactions. Here, we report on the photophysical characterization of two perylene diimides substituted in the bay position with bulky fused aromatic rings, naphthalene (PDI-1) and acenaphtene (PDI-2). Then we show that both PDIs can be used to prepare solution-processed red-emitting OLEDs, taking advantage of their highly improved solubility and emission efficiency in the solid state. We demonstrate that this class of molecules are good candidates for red and deep red emissive OLEDs.

Experimental

Materials and methods

The synthetic procedures for the preparation of PDI-1 and PDI-2 are described elsewhere [24]. The electroactive polymer poly[(9,9-dioctylfluorenyl-2,7-diyl)-alt-co-(1,4-benzo-{2,1,3}-thiadiazole)] (F8BT) was purchased from Sigma-Aldrich.

UV-Vis absorption spectra were obtained with a Perkin Elmer Lambda 900 spectrometer. Photoluminescence (PL) and electroluminescence (EL) spectra were obtained with a SPEX270M monochromator equipped with a liquid N2 cooled charge-coupled device, in case of PL exciting with a monochromated 450WXe lamp. The spectra are corrected for the instrument response. PL quantum yields (QYs) of toluene solutions were obtained by using Rhodamine 6G as the reference. PL QYs of casted films were measured using a home-made integrating sphere according to the procedure reported elsewhere [25]. These results are not reported for spin coated films due to the low intensity of the emission for thin samples.

Device Fabrication and Characterization

To investigate the performance of PDIs we fabricated OLEDs with two structures, ITO/PEDOT:PSS/PDI/Ba/AI and ITO/PEDOT:PPS/PVKc/PDI/PF-PEG/Ba/AI, where PF-PEG represents a polyfluorene-based cathode interlayer [26].

For the realization of OLEDs, indium tin oxide (ITO) covered glass substrates were cleaned ultrasonically in deionized water, acetone and 2-propanol. Subsequently, PEDOT:PSS (Heareus Clevios P VP AI 4083) water solution was spin coated on substrates through 0.45 μ m nylon filter, creating hole injecting layer (HIL) with thickness of 45 nm. Then, substrates were annealed at 100 °C for 10 min in nitrogen filled glove box. In all types of devices, PDI-1 and PDI-2 were deposited from toluene solutions with a concentration of 10 mg/ml. In case of simple devices, 80 μ l of PDI solutions were deposited on a substrate and rotated at 800 rpm, forming an emitting layer of 75 nm. In case of optimized OLEDs, 80 μ l of a crystalline poly(N-vinylcarbazole)(PVKc, Sigma-Aldrich, 10⁶ g/mol)

chlorobenzene solution with a concentration of 10 mg/ml was spin coated on substrates with PEDOT:PSS and annealed at 100°C for 30 min, creating a 55 nm thick hole transporting layer (HTL). Then 80 μ l of a PDI solution was deposited in the same way as for simple type OLEDs. Subsequently, 20 μ l of PF-PEG methanol solution was deposited on device rotating at 4000 rpm, creating a 15 nm electron injecting layer (EIL). Construction of all devices was concluded with vacuum evaporation of cathode, consisting of barium (7 nm) and aluminum (100 nm). Electrical characterization of the devices was performed in nitrogen atmosphere with a Keithley 2602 source-meter combined with calibrated photodiode with Lambert source assumption.

The thickness of organic stack was 120 nm in simple OLEDs and 190 nm in the optimized devices, measured by Bruker Dektak XT profilometer.

Results and discussion

Synthesis and characterization

The structures of the perylene diimide fluorophores used as emitters in this study are shown in Figure 1. PDI-1 and PDI-2 were prepared according to literature procedure [24]. These molecules are soluble in common organic solvents and characterized by high thermal stability (Figure ESI-1).





Suzuki coupling reaction of 1,7-dibromo-N,N'-di-(10-nonadecyl)perylene-3,4,9,10-tetracarboxylic diimide with naphthalene-1boronic acid and acenaphthene-5-boronic acid gave PDI-1 (78% yield) and PDI-2 (63% yield), respectively.

The electrochemical properties were investigated by cyclic voltammetry (see Figure ESI-2). Both PDIs undergo two chemically reversible reductive processes. The half way reduction potentials $E_{red}^{1/2}$ for PDI-1 and PDI-2 are -0.67 eV and -0.77 eV, respectively.

The HOMO-LUMO energy levels (see Figure 3) were determined using the empirical formulas E_{LUMO} =-e($E_{red}^{1/2}$ +4.4V) and E_{HOMO} = E_{g}^{opt} + E_{LUMO} . The optical energy band gap E_{g}^{opt} was calculated from the onset of the absorption in the solid state, being 2.06 eV for PDI-1 and 1.92 eV for PDI-2. The calculated values for HOMO-LUMO energies using density functional theory (Figure ESI-3) are in line with the experimental results [24].

Photophysical properties

As shown in Figure 2, the two molecules dissolved in toluene solution display unstructured emission spectra, quite different from the well-structured profiles of unsubstituted perylene diimides, and mirrorless absorption-emission spectral profiles. For both PDI-1 and PDI-2, the spectroscopic features can be correlated to the twisted structure of the perylene core, due to the presence of sterically hindered fused aromatic substituents.

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Figure 2. Optical absorption spectra of PDI-1 and PDI-2 in toluene solutions (dotted lines) and spin coated films (thin solid lines) and PL spectra of toluene solutions (dashed lines) and spin coated films (thick solid lines).

The Stokes shift, usually small (0.06 eV) for the unsubstituted perylene diimide, increases to 0.28 eV with the naphthalene substituent (PDI-1) and to 0.38 eV when the bulkier acenaphtene substituent (PDI-2) is used. Moreover, the absorption profile of the latter broadens and displays two main bands. In Table 1 the photophysical properties of the two compounds in solution and solid state are reported. As shown in Figure 2, the absorption and the PL spectra of both molecules as spin coated films are slightly red shifted compared to those in solutions, which indicates a weak intermolecular interaction in the solid state.

Table 1. Optical properties of **PDI-1** and **PDI-2** in toluene solution (1.0x10⁻⁴M) and in the solid state

	Solution			Film			
	λ_{abs}	λ_{em}	QY	$\lambda_{abs}{}^a$	λ_{em}^{a}	QY ^b	
	(nm)	(nm)	(%)	(nm)	(nm)	(%)	
PDI-1	543	620	98	550	640	38	
PDI-2	465, 558	672	54	480, 565	690	23	

^aSpin coated films; ^bCast films from toluene solution

Typically, perylene diimides possess excellent emission properties in solution, while the emission efficiency sharply decreases upon molecular aggregation [14]. The introduction of the naphthalene substituent (PDI-1) reduces the ACQ phenomenon providing a film with red emission and a PL QY of 38%, highly desirable for optoelectronic applications. The substitution with the more planar acenaphtene substituent (PDI-2) is able to further shift the film emission to the deep red region (690 nm), maintaining a reasonably high PL QY of 23%.

OLEDs

The efficient red and deep red photoluminescence and processability of the PDIs motivated us to prepare red emissive OLEDs by using fully solution-processing techniques.

In order to fabricate red emissive OLEDs employing the two PDIs, we followed two different strategies, namely the dispersion of the dyes into an electroluminescent polymer and the use of the dyes as bulk films. We therefore dispersed PDI-1 and PDI-2 at 5% concentration in a matrix of poly[(9,9-dioctylfluorenyl-2,7-diyl)-alt-co-(1,4-benzo-{2,1,3}-thiadiazole)]

(F8BT), a widely used electroluminescent polymer whose optical properties fulfil the request for resonant energy transfer (RET) to the dye (see Figure ESI-4).

As expected, the polymer emission is almost completely suppressed in F8BT/PDI blends as a consequence of a nearly complete resonant energy transfer from F8BT to the dye. Nevertheless, the PL QYs of the PDIs dispersed in the polymer are quite low (see Table ESI-2), even lower with respect to those of the dyes in solution and as bulk films. This observation suggests that charge transfer processes between the polymer and the PDIs are very efficient and act as PL quencher [24]. The poor emissive performances of the blends suggest that, for the realization of light emitting devices, the use of PDIs films with reduced ACQ phenomena is preferable than blending them in an electroactive matrix.

Accordingly, we prepared OLEDs in which the emitting layers are bulk PDIs films, by using fully solution-processing techniques. Both perylene diimide molecules showed good film forming properties (Figure ESI-5). The devices with simple single layer architectures (ITO/PEDOT:PSS/PDIs/Ba/AI) show an EL with onset voltages of about 6 V and quite low efficiency, probably due to unbalanced charges.



Figure 3. Electroluminescence spectra of PDI-1 and PDI-2 for optimized (ITO/PEDOT:PSS/PVKc/PDI/PF-PEG/Ba/AI) devices at 10 V and 8 V, respectively.

We further optimized both OLEDs with structures of ITO/PEDOT:PPS/PVKc/PDI/PF-PEG/Ba/AI by using additional interlayers while maintaining a fully solution deposition for the preparation of the device active layers (Figure ESI-6). As hole transporting layer, the well established, commercially available, high molecular weight poly-(9-vinylcarbozole) (PVK) was added over the PEDOT:PSS. To make PVK insoluble a thermal annealing was afterwards performed [26]. After the deposition of the emitting layer, we spin coated an alcohol soluble fluorene-based polymer with polar functionalities PF-PEG (poly[(2,7-(9,9'-dioctyl))fluorene)-*alt*-2,7-(9,9'-bis(5''-poly(ethylene oxide) pentyl) fluorene)] as EIL [27]. As a result, the integrity of the emitting layer is preserved because MeOH is a non-solvent for perylene diimides based materials.

The EL spectra of the optimized OLEDs are shown in Figure 3 and are well reproducing the PL spectra. The devices with PDI-1 and PDI-2 emit red- and deep-red light with emission bands at λ =649 nm and λ =693 nm and CIE coordinates of (x=0.66, y=0.33) and (x=0.69, y=0.29). Although the optimized devices are in both cases much thicker than the single layer OLEDs, the turn on voltage decreases from 6-7V to 4V, indicating

DOI: 10.1039/C6RA10467C

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improved balanced charge injection and transport. Moreover, the PVK and PF-PEG layers effectively confine the charge recombination zone far from the electrodes (see the energy levels in the inset of Fig. 3) thus reducing quenching processes. In addition, the EL spectra remain stable over a wide range of driving voltages, confirming that the charge radiative recombination occurs only within the PDIs layer (Figure ESI-7).

Table 2. Optoelectronic properties of PDI-1 and PDI-2

Structuro	V	FI	EOE <i>a</i>			Ь
Structure	V _{on}	ELpeak	EQE _{max}	LEmax	PEmax	Lmax
	[V]	[nm]	[%]	[cd/A]	[lm/W]	[cd/m ²]
ITO/PEDOT:PSS/	6	635	0.0022	0.0011	0.0003	2.6
PDI-1/Ba/Al	0					
ITO/PEDOT:PSS/	7	675	0.00077	0.00095	0.00032	3.5
PDI-2/Ba/Al	,					
ITO/PEDOT:PSS/						
PVKc /PDI-1/	4	649	0.238	0.065	0.017	141
PF-PEG/Ba/Al						
ITO/PEDOT:PSS/						
PVKc/PDI-2/PF-	4	693	0.638	0.0375	0.014	46
PEG/Ba/Al						

 a Total maximal external quantum efficiency, b maximal value for forward emission

As a consequence of these effects, the EQE increases by two orders of magnitude for the optimized OLEDs. The PDI-2 based device shows the best efficiency reported so far in the deep red region (emission at 693 nm, CIE x=0.69, y=0.29) for a fully solution-processed OLED based on a simple PDI-based emitter.

Conclusions

In summary, we have presented the optical and electroluminescence properties of two perylene diimide molecules substituted in the bay positions with sterically hindered fused aromatic units. The presence of naphthalene or acenaphtene moieties on the perylene core, reduces the ACQ in the solid state, preserving a rather good conjugation and emissive properties. This feature, combined with their high stability, suitable HOMO-LUMO energy levels, good filmforming properties and emission in the red/deep red region, were exploited for OLED applications. We show that under optimized conditions, fully solution processable OLEDs employing bulk films of PDI as emissive layers, provide deep red emission with the best efficiency reported so far for devices based on perylene diimide derivatives.

Acknowledgements

This work has been supported by NANOSILK CNR-SAS Joint Research Project, by Slovak Grant Agency for Science contract No. 2/0142/14 and Accordo Quadro between Regione Lombardia and CNR – Cluster Project 'Energy' n°17348. The authors would like to thank W. Porzio for the XRD measurements.

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GRAFICAL ABSTRACT

Perylene Diimide Derivatives as Red and Deep Red-Emitters for Fully Solution Processable OLEDs

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