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Chiral oligothiophenes with remarkable circularly polarized luminescence and electroluminescence in thin films

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Abstract: We report the first observation of circular polarized electroluminescence (CPEL) in thin films of self-organized oligothiophenes. Four new 1,4-phenylene and 9*H*-carbazole-based oligothiophenes were *ad hoc* designed to ensure efficient spontaneous formation of chiral supramolecular order. They were easily synthesized and their chiroptical properties in thin films were measured. Circularly polarized luminescence (CPL) spectra revealed g_{lum} in the order of 10^{-2} on a wide wavelengths range, originating from their self-organized chiral supramolecular organization. These molecules have reasonable properties as organic semiconductors and for this reason they can constitute the active layer of circularly-polarized organic light-emitting diodes (CP-OLEDs). Thus, we could investigate directly their electroluminescence (EL) and CPEL, without resorting to blends, but rather in a simple multilayer device with basic architecture. This is the first example of a CP-OLED with active layer made only of a small organic compound.

Circularly polarized luminescence (CPL) is the differential emission of left- (I_L) and right-handed (I_R) circularly polarized (CP) light, observed for a non-racemic chiral system, usually after photoexcitation with non-polarized light. It is conveniently expressed in terms of luminescence dissymmetry factor g_{lum} , defined as

$$g_{lum} = 2 \times (I_L - I_R) / (I_L + I_R) = 2 \times CPL / I \quad (1)$$

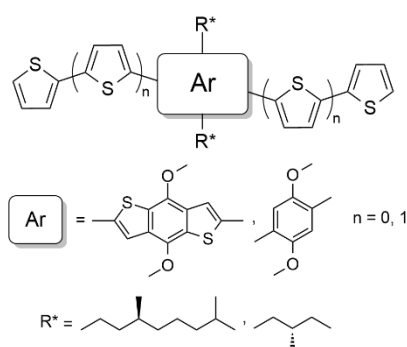
where I is the total emission intensity (*i.e.* the sum of I_L and I_R). For chiral organic π -conjugated molecules g_{lum} hardly exceeds the order of $10^{-4}/10^{-3}$, due to the strong electric dipole-character of their electronic transitions.^[1] However, their self-assembly into chiral aggregated structures may increase g_{lum} values up to more than one order of magnitude.^[2] A similar phenomenon can also occur in thin films, since chiral supramolecular architectures in the solid state can act as efficient source of CP light. Although some chiral π -conjugated systems with promising CPL in thin films have been found,^[3] the field still lacks a systematic search and subsequent development.

Moreover, CPL measurements of thin films require special care in order to avoid the occurrence of false signals.^[4] The most relevant source of artifacts comes from the linear polarization (LP) of emitted light, interfering with the unavoidable residual birefringence of the photoelastic modulator (PEM) of spectropolarimeters;^[5] sample anisotropies can be recognized, quantified and possibly corrected, by rotating and flipping the sample with respect to the instrumental optical axis. A further source of artifacts is due to photoselection, which is strictly related to the experiment geometry and to the polarization of the exciting radiation.^[5] More recently, evidence of the interference between luminescence anisotropy and linear birefringence has been reported, which leads to CPL with the observation of non-reciprocal CPL.^[6]

In recent years CPL has found appealing applications in organic optoelectronics and photonics.^[7] In order to obtain circularly polarized electroluminescence (CPEL), the final emitter must be chiral and non-racemic, which can be obtained with several strategies.^[8] In analogy to g_{lum} for CPL, one may define the electroluminescence dissymmetry factor g_{EL} for CPEL measurement. The use of small organic molecules as organic semiconductors in OLED active layers has gained substantial interest and among them oligothiophenes have a primary role.^[9] Although CPEL was described in various classes of chiral π -conjugated systems,^[8] to the best of our knowledge no examples of CP-OLEDs based on chiral oligothiophenes have been reported to date.

Recently, we studied the electronic circular dichroism (ECD) properties of thin film samples for a family of benzo[1,2-*b*:4,5-*b'*]dithiophene and 1,4-phenylene-based oligothiophenes.^[10] Unfortunately, all of them revealed no emission as thin films due to aggregation-induced quenching (ACQ), thus preventing the collection their CPL spectra. In order to overcome this problem, here we design a set of new chiral π -conjugated oligomers **1–4** (Figure 1) by modifying the original chemical structure following two different approaches: i) introducing alkynyl moieties between the aromatic central ring and the oligothiophenyl units; ii) using the

Previous works:



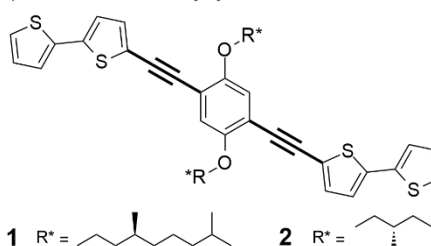
- Outstanding ECD in thin film

but

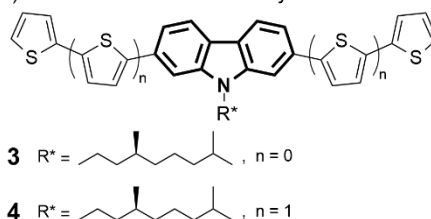
- No emission as thin film → no CPL

This work:

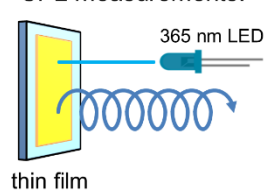
a) introduction of alkynyl moieties:



b) use of 9H-carbazole moiety:



CPL measurements:



CPEL measurements:

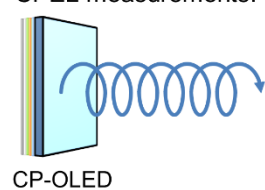


Figure 1. Scope of the work: four new structurally related 1,4-phenylene and 9H-carbazole-based oligothiophenes **1-4** were *ad hoc* designed and synthesized, in order to study their CPL and CPEL features in thin films.

9H-carbazole (CBZ) moiety as central core. Triple bonds as π -spacers not only red-shift the absorption spectrum: they ensure π -conjugation regardless of the dihedral angle between adjacent aromatic groups linked to it;^[11] on the other hand, CBZ-based compounds have appealing optical properties in the solid state, high charge carrier mobility and low redox potential, together with high thermal and photochemical stability.^[12]

Attracted by a possible application as active layers in CP-OLEDs, here we report the chiroptical investigation in emission of our new chiral systems **1-4** as thin films, studying their CPL and CPEL features: to the best of our knowledge, we describe the first observation of CPEL in thin films of chiral oligothiophene-based systems (Figure 1).

The synthetic pathway for the preparation of compounds **1-4** is reported in Supporting Information (Scheme S1). In their isolated form, these molecules can be described as an intrinsically achiral chromophore, decorated with one or two chiral alkyl

chain(s): in these conditions, one cannot expect prominent chiroptical features.^[1, 13] However, the aggregation into chiral supramolecular structures upon thin film deposition can be responsible of large ECD^[14] and CPL signals.^[2]

With such compounds in hand, we prepared thin films, by depositing their solutions onto transparent glass plates by spin coating (SC). All the film samples (thickness ~ 100 nm) macroscopically appeared homogeneous and semi-transparent. Actually, a more detailed morphological characterization via field-emission scanning electron microscopy (FE-SEM) analysis revealed marked differences between 1,4-phenylene-based and CBZ-based compounds: **SC-1** and **SC-2** (Figures S1a-b) showed distinct grain-like domains (5–10 μm size), each of them consisting of thousands of irregular rod-like nanostructures; sample **SC-3** (Figure S1c) displayed instead a more homogeneous texture, with recurring triangular-type structures (about 1 μm size); finally, **SC-4** thin film (Figure S1d) showed a more fibrous texture with

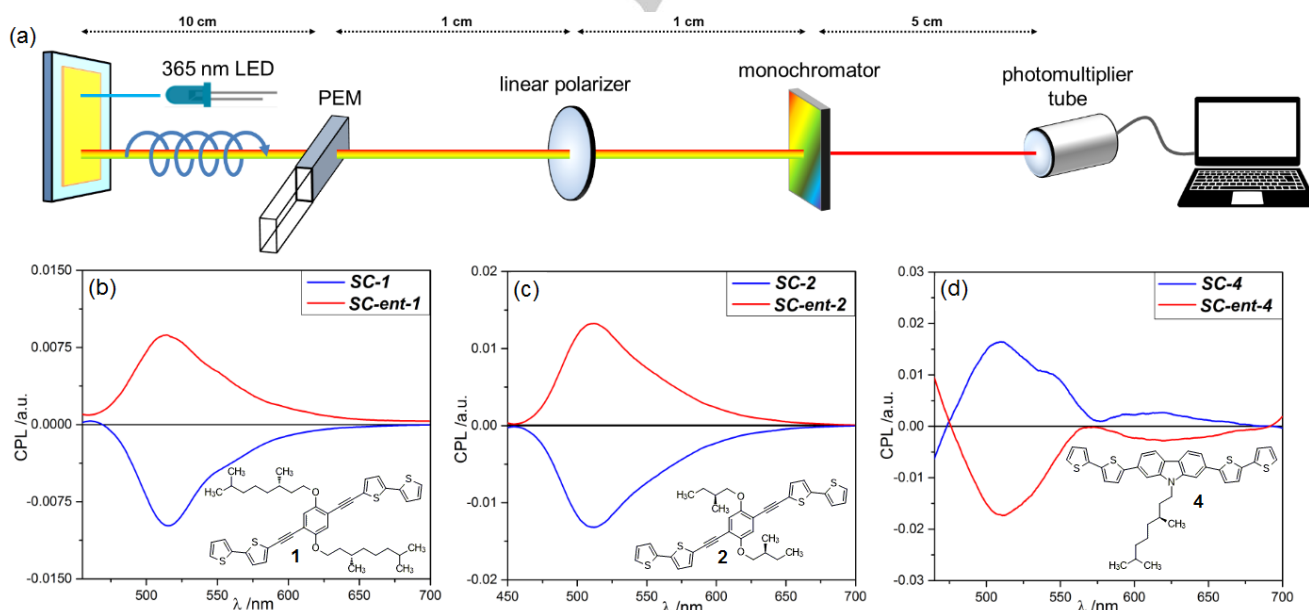


Figure 2. (a) Experimental set-up employed for CPL measurements of thin films with a 0° excitation geometry. (b-d) CPL spectra (normalized with respect to maximum emission) recorded for chiral oligomers **1,2,4** (blue lines) and their opposite enantiomers **ent-1,2,4** (red lines) as spin-coated thin films. Excitation wavelength: 365 nm.

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several μm -long filaments departing from a common core, where each filament is obtained by the helical winding of hundreds of fibers having smaller diameter. A low degree of order was detected in all the thin films by means of X-ray diffraction (XRD) technique (Figure S2), with weak peaks barely observed for each sample.

Their (chiro)optical properties in absorption are described in detail in the Supporting Information (Figures S3-S4 and Table S1). The introduction in **1-2** of alkynyl moieties between the central ring and the bithienyl units led to a substantial increase in thin film photoluminescence (Figures S5a-b) compared to the parent compounds described in our previous works,^[10] which displayed no emission. Similarly, also **SC-3** and **SC-4** displayed strong photoluminescence (Figures S5c-d). This made all of them suitable candidates for the study of CPL.

In the set-up for CPL measurements (Figure 2a) each sample was illuminated from the same direction of collection of the emitted light (ideally, 0° geometry), putting a 365 nm LED source at the minimum angle $\alpha \approx 10^\circ$ with respect to the optical axis. This geometry may be itself a source of LP of the emitted light, but to make sure that it does not affect the CPL measurement, not only we measured LP through the same instrument (by operating it at twice the PEM frequency), but we also tried different orientations of the LED, by changing α and by moving the LED on an arch around the optical axis. For each compound, we performed these tests on thin film samples of both enantiomers for **SC-1**, **SC-2**, and **SC-4** (Figures 2b-d), observing in all cases the required symmetry relationships in the CPL results, confirming that for our films this geometry is suitable to record *true* CPL. Moreover, the large Stokes shift of samples **SC-1**, **SC-2**, and **SC-4** ensures that we can exclude self-absorption artifacts due to circular dichroism.^[15] Unfortunately, **SC-3**, which has the most blue-shifted emission, did not appear suitable for our instrumental setup and because of excessive back scattering of the exciting radiation, and for these reasons it was discarded.

Sample **SC-1** displayed a relatively modest CPL (Figure 2b) compared to the structurally similar oligomer **2**, which instead is characterized by a maximum g_{lum} of $-1.3 \cdot 10^{-2}$ (Figure 2c). The fluorescence spectrum of **SC-4** (Figure S5d) exhibited a large band centered at 513 nm, together with a prominent shoulder at ~ 550 nm tentatively attributed to a vibronic component. This trend was also reflected in the CPL spectrum (Figure 2d), showing a strong positive band between 475 and 575 nm, with maximum at 510 nm ($g_{lum} = 1.7 \cdot 10^{-2}$), and a shoulder at ~ 550 nm; a further broad weak positive band was found at longer wavelengths, between 575 and 675 nm.

In order to investigate a possible application of our oligothiophenes as chiral emitting materials, first we performed preliminary EL measurements on *standard* OLED devices with a very basic architecture (Figure S6): as anode we used a glass/ITO plate, covered with PEDOT:PSS; our π -conjugated oligomers were then deposited as neat material by spin coating (~ 100 nm of thickness); finally, a Ca cathode (30 nm) with an Al protective layer (200 nm) was selected because of its good performance in many highly efficient OLED devices.^[16]

In these conditions, only **SC-4** exhibited EL: this could be related to the fact that CBZ moiety is an excellent hole-transporter, with better hole injection properties compared to the 1,4-phenylene core of oligomers **1-2**.^{[12a],[12b]} The current density vs. voltage (J - V) characteristics (Figure S7a) showed that the OLEDs switched on at ~ 7 V; however, they were driven at $4.5 \cdot 10^{-4}$ A cm^{-2} (corresponding to ~ 9 V) for EL measurements, carried out by collecting the emitted light from the transparent glass/ITO surface in perpendicular direction. The device luminance (Figure S7b) was collected simultaneously during the transfer characteristics measurements, showing a similar trend as the J - V curve, while the external quantum efficiency (EQE) was found below 0.001% (Figure S7c). EL spectrum (Figure S7d) showed a profile quite similar to photoluminescence (Figure S5d), although the band is slightly red-shifted (maximum emission at 536 nm) and with a large tail at long wavelengths, due to the presence of some oxidized

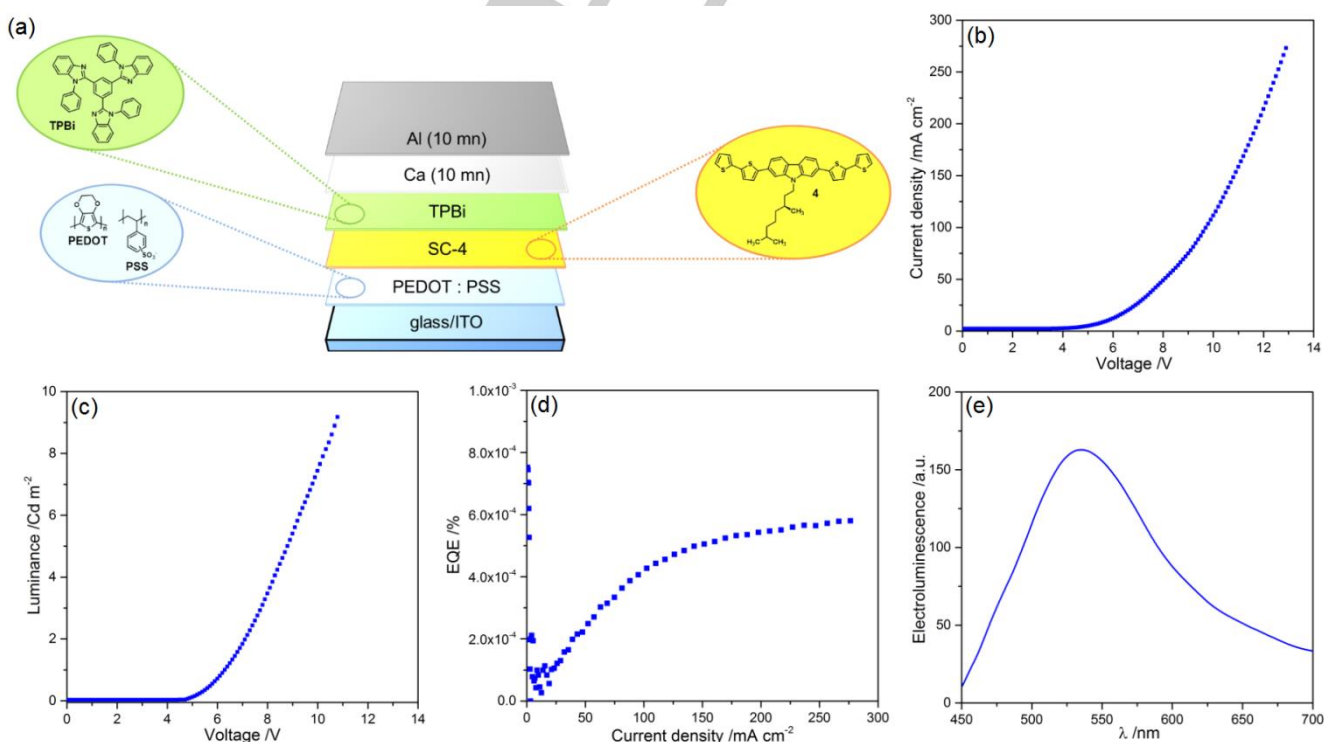


Figure 3. (a) Architecture, (b) representative current density vs. voltage (J - V) curve, (c) luminance vs. voltage curve, (d) external quantum efficiency (EQE) vs. current density curve and (e) EL spectrum of *multilayer* OLED devices based on **SC-4** as active layer.

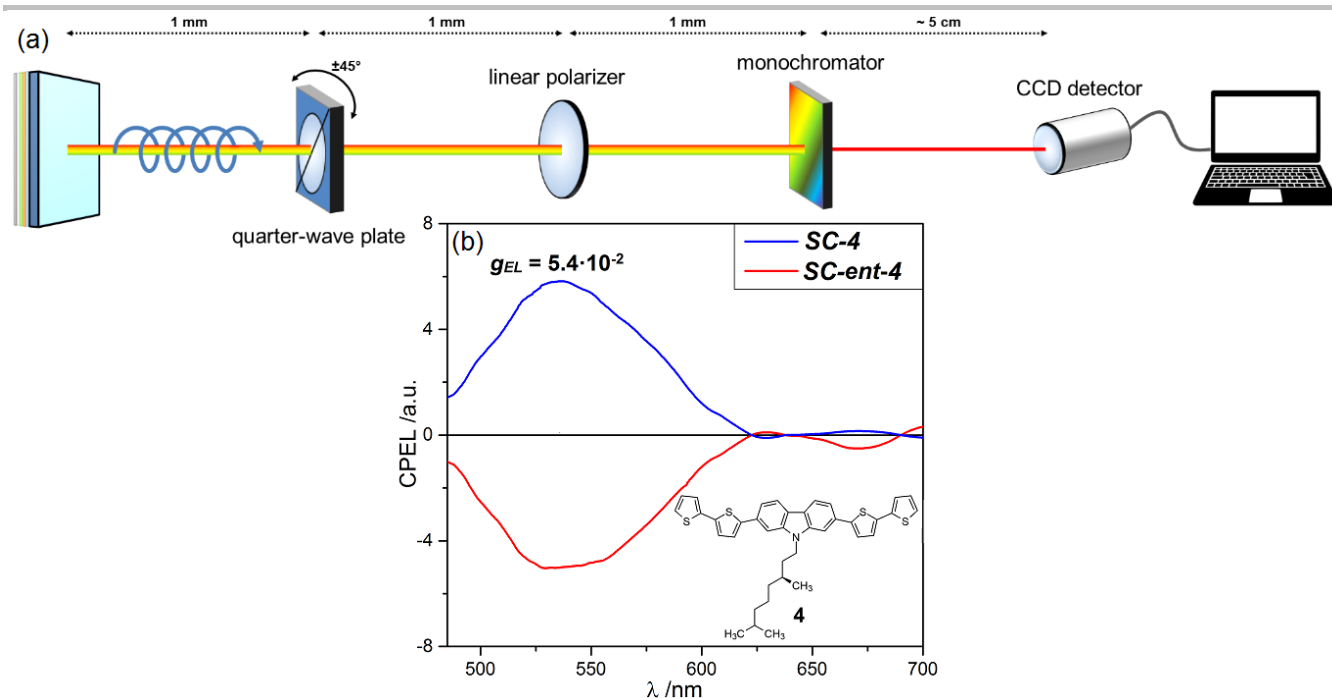


Figure 4. (a) Experimental set-up employed for CPEL measurements of *multilayer* CP-OLED devices. (b) CPEL spectra of *multilayer* CP-OLED devices based on spin-coated thin films of **4** (blue line) and its opposite enantiomer **ent-4** (red line) as active layer.

centers acting as charge traps and emitting at lower energy, or to a preferential charge migration to lower energy sites before exciton formation.

The configuration of *standard* OLEDs used for EL measurements of **SC-4** includes a reflecting metal top electrode, which redirect in the “forward” direction also photons emitted in the opposite direction (*i.e.* with respect to the transparent glass/ITO substrate) but with a reversal of the handedness of their circular polarization, resulting in a substantial decrease of g_{EL} value despite the intrinsic emission of the active layer being strongly polarized.^[17] In CP-OLEDs this would be highly detrimental, therefore the reduction of the cathode thickness to obtain a semi-transparent surface is fundamental. Following this strategy, we fabricated a new set of devices with a very thin cathode, *i.e.* 10 nm Ca covered by 10 nm of Al. Unfortunately, these new devices revealed no EL: cathode thickness is a very critical parameter in determining the devices performance, and its excessive reduction may involve a modification of their optical and electrical features. We successfully addressed this issue by introducing a *cathode interfacial layer* (CIL). It was able not only to improve the OLED performance by promoting the injection of electrons into the active layer, but also to partially absorb the light directed to or reflected by the back electrode.^[6, 18] We selected 1,3,5-tris(1-phenyl-1*H*-benzo[d]imidazol-2-yl)benzene (TPBi), having good electron-transporting and hole-blocking properties.^[19] Furthermore, TPBi showed orthogonal solubility with respect to **4**: the first is soluble in CH₃OH, the second in CHCl₃.

In this way, we fabricated *multilayer* devices (Figure 3a): on the top of PEDOT:PSS covered glass/ITO substrate, a **SC-4** layer (~ 100 nm of thickness) was deposited; it was then covered with a TPBi layer, prior to the thin cathode (10 nm Ca covered by 10 nm of Al). Their *J-V* characteristics (Figure 3b) showed that OLEDs switched on at ~ 8 V, but with higher current density (one order of magnitude) compared to the previous *standard* devices, thus confirming the improvement in electrical properties due to the presence of TPBi. The luminance curve (Figure 3c) is significantly higher compared to that of *standard* device: this is mainly related to the increased

electrical current passing through the device, also thanks to the TPBi layer. However, maximum EQE was found again below 0.001% (Figure 3d): actually this is not surprising, since such devices have not been optimised for high external quantum efficiency values. EL measurements, carried out in this case at $4 \cdot 10^{-3}$ A cm⁻² (corresponding to ~ 9 V), exhibited a profile (Figure 3e) very similar to the one recorded for *standard* OLEDs: a large emission band with maximum at 534 nm and a pronounced baseline drift at long wavelengths. In order to verify the lifetime stability of our devices, EL measurements were repeated 5 times for each pixel.

Finally, we measured the CPEL of these *multilayer* devices using a quarter wave retarder and a linear polarizer prior to a CCD detector: by rotating the easy axis of the quarter wave plate by $\pm 45^\circ$ with respect to the linear polarizer, the I_L and I_R components of the emission were alternatively acquired (Figure 4a). By subtracting the I_L and I_R spectra, the CPEL spectra were obtained, while g_{EL} values were calculated according to the definition reported in Eq. (1). **SC-4** (Figure 4b, blue line) showed a wide positive band between 480 and 650 nm, with maximum intensity at 536 nm and $g_{EL} = 5.4 \cdot 10^{-2}$. The sign of CPEL band was the same of that previously found in CPL spectrum (Figure 2d), while the dissymmetry factor g_{EL} appeared increased (about three times larger than g_{lum}). This neat increase of the CPEL dissymmetry factor g_{EL} compared to the corresponding CPL value g_{lum} is a very interesting aspect, which has been previously reported in the literature in few cases.^[3c, 20] These measurements were repeated 5 times for each pixel of four different devices; furthermore, we recorded CPEL of devices based on the opposite enantiomer **ent-4**, fabricated under the same experimental conditions (Figure 4b, red line). In this way we excluded the occurrence of parasitic signals due to LP and/or to birefringence of any component in both OLEDs and detection apparatus, confirming the total reproducibility of our results.

To the best of our knowledge, this *multilayer* device represents the first example of CP-OLEDs based on chiral oligothiophenes as organic semiconductor and at the same time the CPL-emitter. But, more importantly, this is the first CP-OLED device with active layer

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made of only a single small organic compound as neat material, *i.e.* a very simple architecture compared to other very recent CP-OLEDs based on small organic molecules.^[20c, 21] This marked simplification in the device architecture does not involve an excessive loss of its performance: in fact, the current density and luminance values of our CP-OLED device are not far from those measured for other CPEL emitting devices based on chiral organic small molecules blended in a polymer matrix^[3c] or doped with other small molecules.^[20c, 21]

A few more elements may contribute to the success of this molecular system: the presence of carbazole, which is itself a good hole transporter, and the ethynyl moiety, which ensures efficient π -conjugation, while allowing for relative conformational flexibility and thus preventing the otherwise ubiquitous ACQ. These results represent an important *proof-of-concept* and there is plenty of room for improvement, from the chemical structure of the π -conjugated system to the device architecture.

In conclusion, we have *ad hoc* designed and synthesized four new chiral oligothiophenes, in order to study their emission chiroptical properties in thin films. CPL spectra showed quite large g_{lum} values (in the order of 10^{-2}) on a wide wavelengths range, mainly originating from their chiral supramolecular architectures. These encouraging results led us investigate their possible application as CPEL emitting materials. It is worth emphasizing that in the CP-OLEDs fabrication we always used our π -conjugated oligomers as neat material, which makes fabrication particularly simple, and at the same time it preserves the spontaneous chiral supramolecular organization, which is fundamental in developing strong CPEL signals. The 9*H*-carbazole-based oligothiophene **4** resulted in a *multilayer* CP-OLED device with g_{EL} up to $5.4 \cdot 10^{-2}$, a value significantly higher than the corresponding g_{lum} . This interesting observation may be the result of a somehow different organization of the thin film deposited onto PEDOT:PSS instead of onto glass, or be due to slightly different local geometries, possibly more or less active in photo- vs. electroluminescence. Investigation in these directions is undergoing. Finally, this contribution represents the first observation of CPEL in thin films of chiral oligothiophenes, and more importantly the first example of CP-OLED with active layer made only of a small organic compound.

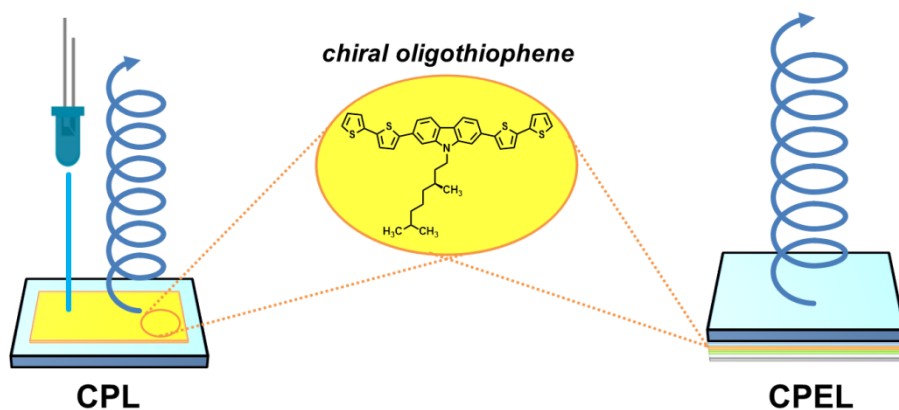
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Keywords: circularly polarized electroluminescence • circularly polarized luminescence • CP-OLED • oligothiophenes • thin films

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Chiroptical properties in thin films of four new chiral oligothiophenes *ad hoc* designed and synthesized were investigated. Remarkable circularly polarized luminescence (CPL) and electroluminescence (CPEL) were observed on a wide wavelengths range, originating from their chiral supramolecular organization. This is the first observation of CPEL in thin films of chiral oligothiophenes.

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