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New derivatives of triphenylene, perylene, and pyrene are described, some of which form columnar mesophases. The absorption spectra are investigated both experimentally and theoretically. The spectra calculated using the density functional tight binding (DFTB) theory are in good agreement with experimental results. The investigated compounds show photoluminescence of violet–blue (triphenylene), yellow–green (pyrene), and orange–red (perylene) colours. In addition, electroluminescence is observed in thin films of these compounds between a positively charged ITO electrode and a negatively charged aluminium electrode. The brightness of the electroluminescence decreases in the order perylene > pyrene > triphenylene. Threshold voltages below 20 V and a luminance up to 100 cd m^{-2} were observed.

1. Introduction

During the last decade, organic light emitting diodes (OLEDs) have been developed to an extent which allows their commercial application for large area displays. Both low molecular mass materials [1] and conjugated polymers [2] can be used to achieve an emission of light at sufficiently low voltages. By means of high quality organic single crystals, even an electrically driven organic laser which operates at low temperatures was developed recently [3].

The least elaborate set-up for investigating electro-luminescent properties consists of a single organic layer sandwiched between a transparent electrode layer (usually indium-tin-oxide, ITO, which exhibits a high work-function and may serve as the positive contact) and a metal electrode (usually a metal with low work function, such as Ca or Al, serving as the negative contact), More efficient electroluminescent devices can be obtained from organic two-layer [1, 4–8] or multilayer systems [9–13]. If two layers of different organic semiconductors are used (figure 1), one being an efficient hole transporting layer (HTL), the other being an efficient electron trans-porting layer (ETL), the zone of the recombination

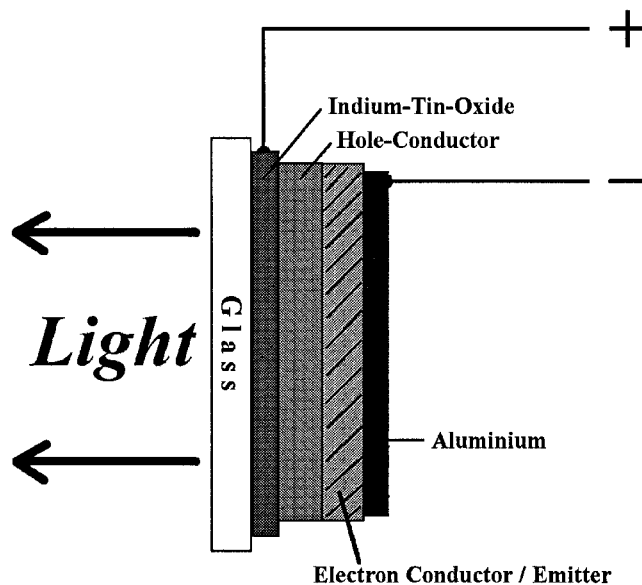Figure 1. Set-up of an organic light emitting diode.

between holes and electrons can be restricted to the HTL/ETL interface, and thus separated from the electrode areas. This reduces the probability of non-radiative recombination of charge carriers. In this two layer arrangement [1, 4], one of the two organic materials must exhibit

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the luminescent properties. Alternatively, a luminescent dye may be incorporated as a third layer between the HTL and the ETL layer [9, 10]. The organic semiconductors used in these systems need to have a high charge carrier mobility. The density of defects which may trap the charge carriers should be as low as possible. An additional issue in improving the performance of OLEDs is the effectiveness of the charge carrier injection at the electrodes. The work function of an electrode should match the electronic states of the neighbouring organic layer appropriately in order to support the charge carrier injection. In addition, a charge injection layer may help to reduce the energy barrier for the charge carrier injection [14].

The development of OLEDs requires suitable electroluminescent dyes, organic semiconductors with high charge carrier mobility and defect-free structures in order to avoid trapping or radiationless recombination of opposite charge carriers. The search for appropriate materials has led to the development and systematic study of new low molar mass organic semiconductors and emitters. In addition to a variety of different hole transporting aromatic amines, diamines or polyamines [1, 4–8, 10], electron transporting triazole or oxadiazole derivatives [4, 5, 7, 9, 10, 15] and luminescent dyes with emission from an aromatic chromophore [1, 5, 7–10], new classes of materials have been taken into account, for example luminescent organometallic rare earth complexes which show a very narrow emission band [13] or electron transporting spiro-compounds which are characterized by very high glass transition temperatures [16, 17]. The use of polymers has been extended from the efficient but insoluble poly(*p*-phenylene vinylene), PPV, [2] to more soluble PPV-derivatives [18, 19], polycarbonate [5], polyfluorene derivatives [20], or polyquinoxalines [21]. Also blends of low molar mass doping materials with polymers, e.g. based on poly(methyl methacrylate) [22] have been tested.

Liquid crystals are of particular interest for organic light emitting diodes [23–30]. Columnar liquid crystals with a suitable molecular structure can exhibit an unusually high charge carrier mobility of the order $0.1\,\mathrm{cm^2\,V^{-1}\,s^{-1}}$ [31]. This value is in the range between amorphous organic layers (typically $\approx 10^{-3}$ to $10^{-2}\,\mathrm{cm^2\,V^{-1}\,s^{-1}}$) and organic single crystals ($\approx 1\,\mathrm{cm^2\,V^{-1}\,s^{-1}}$). The latter materials can have a higher charge carrier mobility, but monocrystalline layers are difficult to grow. Liquid crystals, however, show the ability to self-organize, i.e. defects are healed by annealing the respective compounds in the temperature range of the mesophases. In addition to this self-healing process, some columnar liquid crystals spontaneously show a uniform homeotropic orientation, even on untreated substrates [32].

Thus, liquid crystals are particularly useful for generating uniformly oriented, defect-free layers. Another interesting feature is shown by calamitic, and in particular smectic liquid crystals. Uniform homogeneously oriented nematic or smectic liquid crystals exhibit a linearly polarized luminescence [26–30]. A linear dichroism $I_{\parallel}/I_{\perp}$ of the luminescence intensity up to 25:1 and a luminance of up to $250\,\mathrm{cd\,m^{-2}}$ have been reported recently [30]. Attempts were even made to make use of cholesteric liquid crystals in order to generate circularly polarized light [26], but the latter samples still need significant improvement for consideration in applications. In summary, there is high interest in developing new electroluminescent liquid crystals in order to make use of their high charge carrier mobility, self-assembling properties and/or dichroism of the luminescence.

In the present paper, we report on the luminescent properties of some new columnar derivatives of triphenylene, pyrene and perylene which have been synthesized recently [33]. Here, we present the spectra of absorption, photoluminescence and electroluminescence. The absorption spectra are compared with results from calculations using the time-dependent density functional tight binding (TD-DFTB) theory [34]. For two of the three types of compound, solutions show a remarkable spectral shift of the photoluminescence with concentration. This effect is studied in more detail.

2. Experimental

Here, we present our results on the following derivatives of triphenylene (**1**), pyrene (**2**), and perylene (**3**). Most of the substances show liquid crystalline phases. Their transition temperatures and enthalpies are listed in the table. For details of the synthesis, see [33].

(1)

(2)

(3)
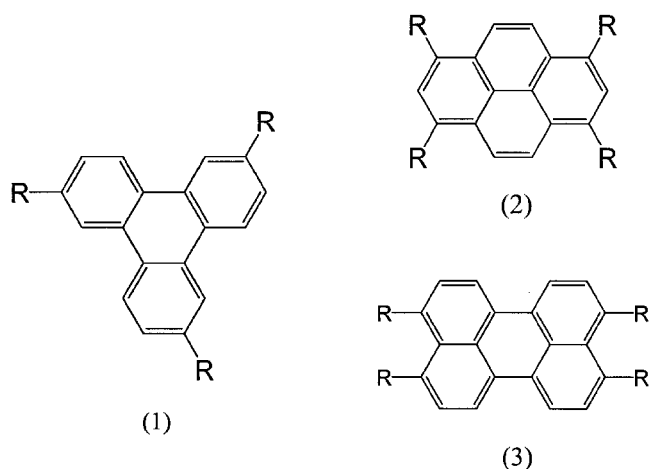
We recorded the absorption spectra by means of a Perkin-Elmer spectrophotometer (model Lambda 19DM) and the fluorescence spectra using a MPF4 spectrophotometer (Perkin-Elmer). In the case of the dilute

Table. Investigated compounds transition temperatures (°C), transition enthalpies (J g^{-1}), and the wavelengths (nm) of maximum absorption and maximum fluorescence for dilute solutions in chloroform and the pure compounds, respectively.

Compound	R	Transition temperatures/°C and enthalpies/J g^{-1} in brackets	Absorption λ_{max}/nm		Fluorescence λ_{max}/nm	
			solution	pure	solution	pure
1a	$CO_2C_2H_5$	Cr−161.24 (63.84)−I−132.66 (19.51)−Col	289	305	384	402
1b	$CO_2C_3H_7$	Cr−137.39 (58.11)−I−81.2 (3.6)−N−66.17 (17.65)−Cr	280	300	384	402
1c	$CO_2C_4H_9$	Cr−86.04 (36.85)−I−58.92 (2.65)−N−40.62 (22.41)−Cr	293	298	384	405
1d	$CO_2C_5H_{11}$	Cr−110.67 (61.04)−I	293	295	383	402
1i	$CO_2CH_2CH(C_2H_5)(C_4H_9)$ rac.	Cr−110.95 (22.15)−Col−123.66 (6.71)−I	290	320	383	404
2a	$CO_2C_2H_5$	Cr−189.8 (45.1)−Col 203.7 (2.5)−I	394	415	414	555
2e	$CO_2C_6H_{13}$	Cr−104.1 (51.2)−I	396	414	413	516
2i	$CO_2CH_2CH(C_2H_5)(C_4H_9)$ rac.	Col−94.5 (10.1)−I	397	415	413	544
3a	$CO_2C_2H_5$	X−134 (16)−X−150 (5)−X−244 (3)−Col$_{hex}$−313 (19.5)−I	443	462	487	606
3b	$CO_2C_3H_7$	Cr−193 (66)−Col$_{hex}$−287 (11.5)−I	443	467	490	602
3c	$CO_2C_4H_9$	Cr−161 (68)−Col$_{hex}$−242 (6.1)−I	444	461	491	586
3d	$CO_2C_5H_{11}$	Cr−114 (73)−Col$_{hex}$−203 (3.8)−I	443	459	491	587
3e	$CO_2C_6H_{13}$	Cr−72 (58)−Col$_{hex}$−177 (3.5)−I	444	454	490	584
3f	$CO_2C_7H_{15}$	Cr−63 (78)−Col$_{hex}$−152 (2.6)−I	444	460	490	584
3g	$CO_2C_8H_{17}$	Cr−62 (29)−Col$_{hex}$−132 (2.3)−I	444	462	490	593
3h	$CO_2C_9H_{19}$	Cr$_1$−44 (35)−Cr$_2$−60 (28)−Col$_{hex}$−100 (1.4)−I	444	457	489	582
3i	$CO_2CH_2CH(C_2H_5)(C_4H_9)$ rac.	Col−240.1 (11.9)−I	472	462	493	603

solutions in chloroform (p.a.), the concentrations of the dyes were approximately 1×10^{-5} mol l^{-1}; for the measurements, 1 cm quartz cells were used. In order to investigate the formation of excimers in the solutions, we investigated the influence of concentration on the luminescence in ethanolic solutions of pyrene, similarly to previous experiments [35, 36]. We then studied the fluorescence of solutions of different concentrations from one of the new compounds. In order to study the pure compounds in the crystalline and in the liquid crystalline states, the compounds were evaporated on a glass substrate at a pressure of approximately 1×10^{-4} mbar; we obtained dye layers with thicknesses of some 100 nm in this way. Prior to the cell preparation, the glass substrates were cleaned in an ultrasonic bath for 15 min using chloroform.

The electroluminescent properties were examined by preparing a thin (≈ 50 nm) layer of the selected compound on indium tin oxide (ITO) coated glass substrates. The material was deposited by thermal evaporation in high vacuum. A thin aluminium electrode (≈ 100 nm) was evaporated on top of the organic layer. Double layer structures (figure 1) were created by spin-coating either a hole conducting layer or a charge carrier injection layer on the ITO substrate. This layer consisted either of *N,N'*-diphenyl-*N,N'*-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine (TPD) or poly-3,4-ethylene dioxythiophene (Baytron P, Bayer AG). TPD was spin-coated from a chloroform solution while Baytron P was spun from an aqueous suspension. In the second step, the emitting layer, consisting of one of the liquid crystalline materials, was evaporated. Finally, the top electrode made of aluminium was deposited. The spectra and the luminance of the electroluminescent samples were measured using a diode array photometer, model PR-650 (Photo-Research). The surface topography of the substrates and the thin organic layers were investigated by atomic force microscopy using a Multimode head and a Nanoscope IIIa controller (Digital Instruments).

3. Results and discussion

The investigated compounds show absorption and photoluminescence in different parts of the visible spectral range (figure 2). The triphenylene derivatives absorb light in the near UV region and show a violet–blue photoluminescence, while the absorption and luminescence of derivatives of pyrene and perylene occur at longer wavelengths. For the same molecular core, differences in the alkyl chain have only a small effect on the wavelength of maximum absorption or emission intensity (see the table). However, the photoluminescence spectra of the pure compounds differ considerably from the spectra of their dilute solutions (figure 2). For all compounds investigated, the photoluminescence maxima of the solutions show a small Stokes shift of about 20 nm with respect to the absorption band. However, the photoluminescence of the pure compounds is red-shifted with respect to the luminescence of the solutions. For the triphenylene com-
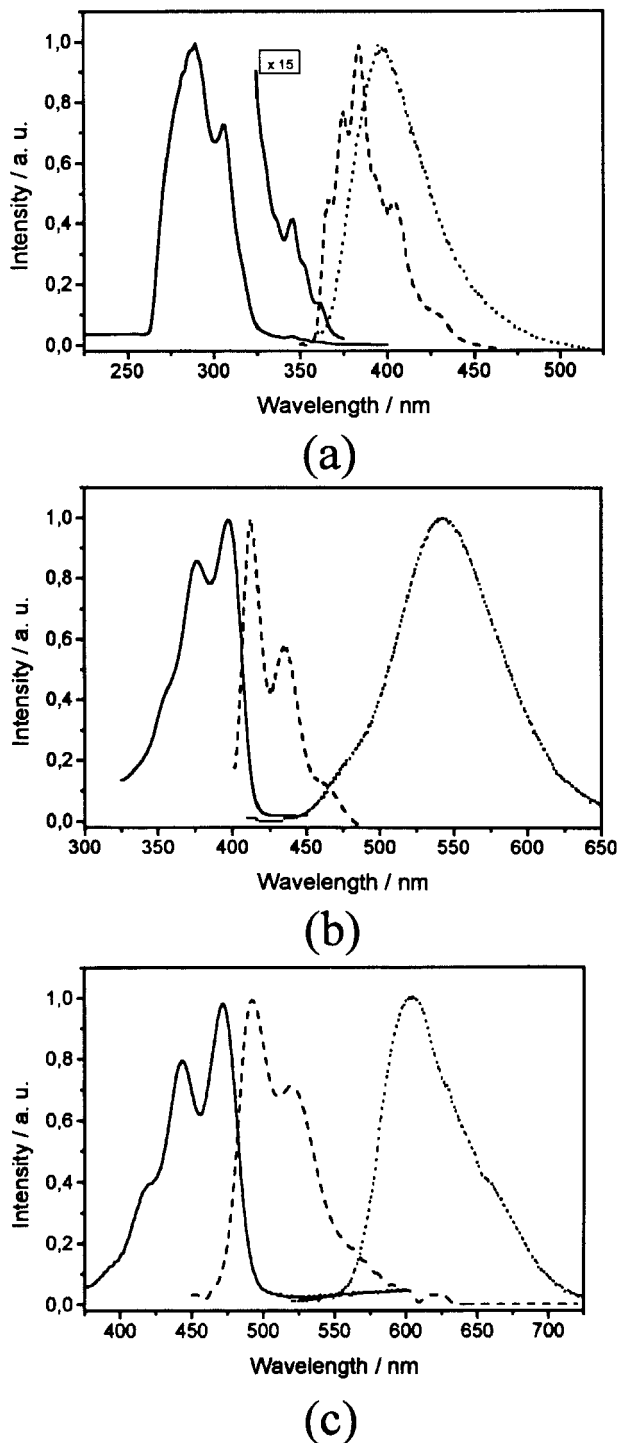
Figure 2. Absorption spectra (——), photoluminescence spectra of solutions (- - -) and photoluminescence spectra of the pure materials (· · ·) for the compounds (*a*) **1i**, (*b*) **2i**, and (*c*) **3i**.
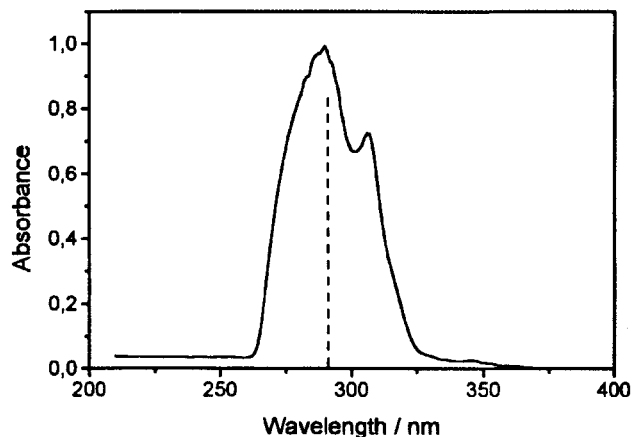
shift to the formation of dimers by excited molecules (excimers) as this is known to occur in other compounds containing the same chromophores. The conditions for this excimer formation are studied in more detail and discussed at the end of this section.
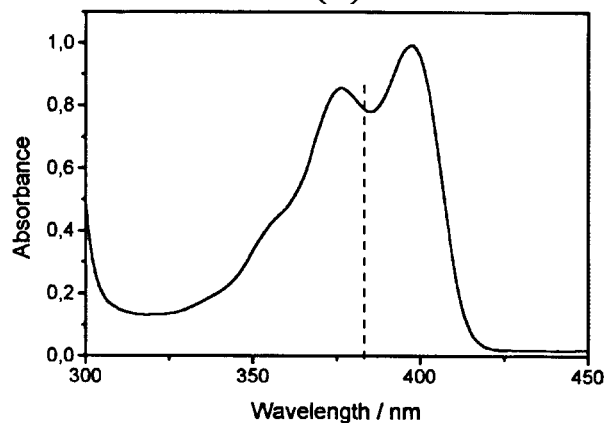
3.1. *Absorption*

With respect to the absorption spectra, we found a good agreement between calculated spectra and our experimental data (figure 3). For perylene compounds, the ground state and the excited electronic state, as well as the vibrational states of the molecules were calculated using the time-dependent density functional tight binding (TD-DFTB) theory [34]. Both the spectral position of the absorption band and the vibrational fine structure are in good agreement with experimental data, figure 3(*c*). In order to test the quality of the DFTB calculations, we also computed the wavelengths of maximum absorption for perylene and another well known perylene derivative, 3,4,9,10-perylene-tetracarboxylic dianhydride (PTCDA). Again, not only the trend of the bathochromic shift caused by substituents, but also the absolute values of the wavelength are in good agreement with our experiment (figure 4). For comparison, calculations were made using the INDO approximation. The latter calculations indicate the direction of the spectral shift with different substituents correctly, but the deviations of the calculated wavelengths from the respective experimental values are rather large (figure 4). For the triphenylene and pyrene compounds, we have so far calculated only the energy difference between the electronic ground state and the electronic excited state. Thus, only the spectral position of the absorption bands (without the vibrational fine structure) can be given, figures 3(*a*) and 3(*b*). Again, these absolute values are in good agreement with the observed absorption bands of the respective compounds.
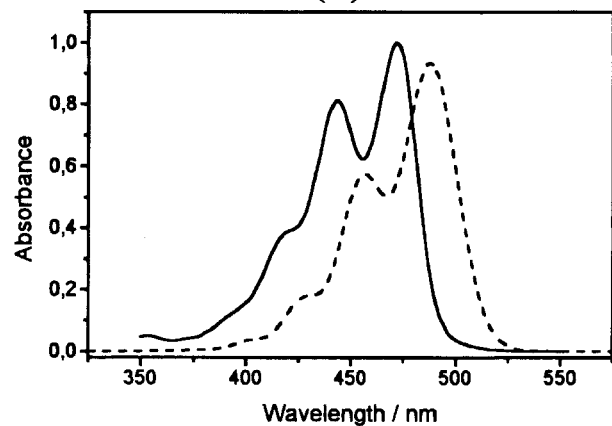
3.2. *Photoluminescence*

The photoluminescence of solutions of the triphenylene compounds leads to a violet–blue colour, while solutions of pyrene and perylene compounds show a blue and a green luminescence, respectively. In contrast, the pure triphenylene, pyrene and perylene compounds display a violet–blue, yellow–green and orange–red luminescence, respectively. Both the red-shift and the lack of vibrational fine structure observed for the pure compounds are typical of the formation of excited dimers (excimers) [37]. We conclude that this excimer formation occurs in the pure compounds, but not in solutions. In order to investigate this behaviour more closely, we studied the dependence of the luminescence spectra on the concentration. In the case of unsubstituted pyrene, both the monomer and the excimer luminescence bands were

pounds, this red-shift amounts to only about 10 nm, but the red-shift is as large as about 100 nm and even more for perylene and pyrene derivatives. We attribute this

(a)

(b)

(c)

Figure 3. Comparison of the experimental absorption spectra (—) with calculations (---) based on the density functional tight binding theory. (*a*) Compound **1i**, (*b*) compound **2i**, and (*c*) compound **3g**—see also the text.

found at a concentration of $0.005 \, \text{mol} \, l^{-1}$ of pyrene in ethanol, figure 5(*a*). If the concentration is varied around this critical value, the wavelengths of these two bands
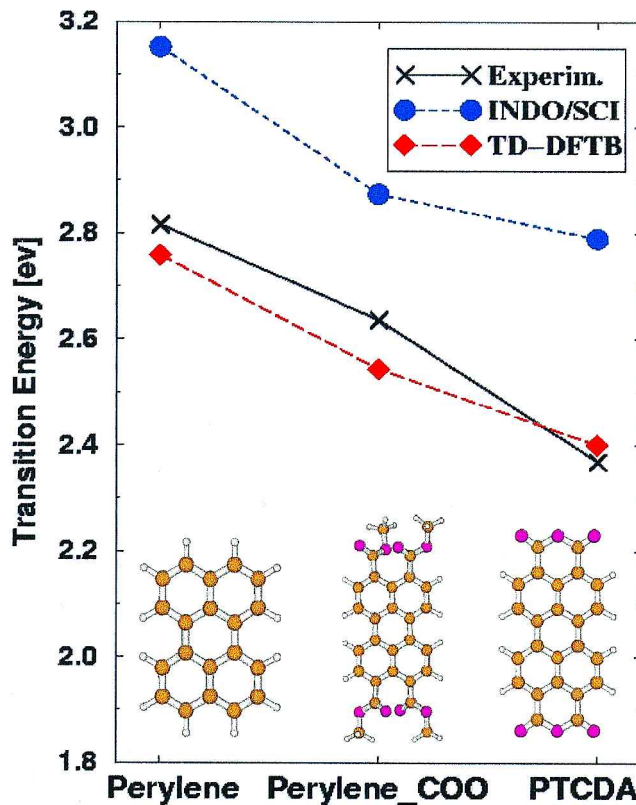

Figure 4. Energy gap between the lowest unoccupied (LUMO) and the highest occupied molecular orbital (HOMO) for unsubstituted perylene, perylene-tetracarboxylic dianhydride (PTCDA), and a perylene-tetracarboxylic ester. (×) Experimental data, (●) calculation using the INDO/SCI method, (◆) calculation using the TD-DFTB method [34].

remain constant, while the intensities of the monomer and excimer band alter, i.e. the excimer band dominates at higher concentration. This change of the two intensities (as opposed to a wavelength shift) is typical of the chemical equilibrium between two compounds. Thus, its observation confirms the assumption of the formation of dimers occurring at higher concentration. The liquid crystalline derivatives are expected to behave in the same way. Indeed, the intensity of the monomer band decreases with increasing concentration in the range investigated up to $10^{-2} \, \text{mol} \, l^{-1}$. However, no excimer band was found. Even for the relatively high concentration of $0.01 \, \text{mol} \, l^{-1}$, the luminescence of the solution is distinctly different from the luminescence of the respective pure compound, figure 5(*b*). This result seems to indicate that substitution with bulky lateral groups hinders the formation of dimers, so that excimer formation can occur only at higher concentrations. The range of higher concentrations was not accessible due to the limited solubility of the compounds.
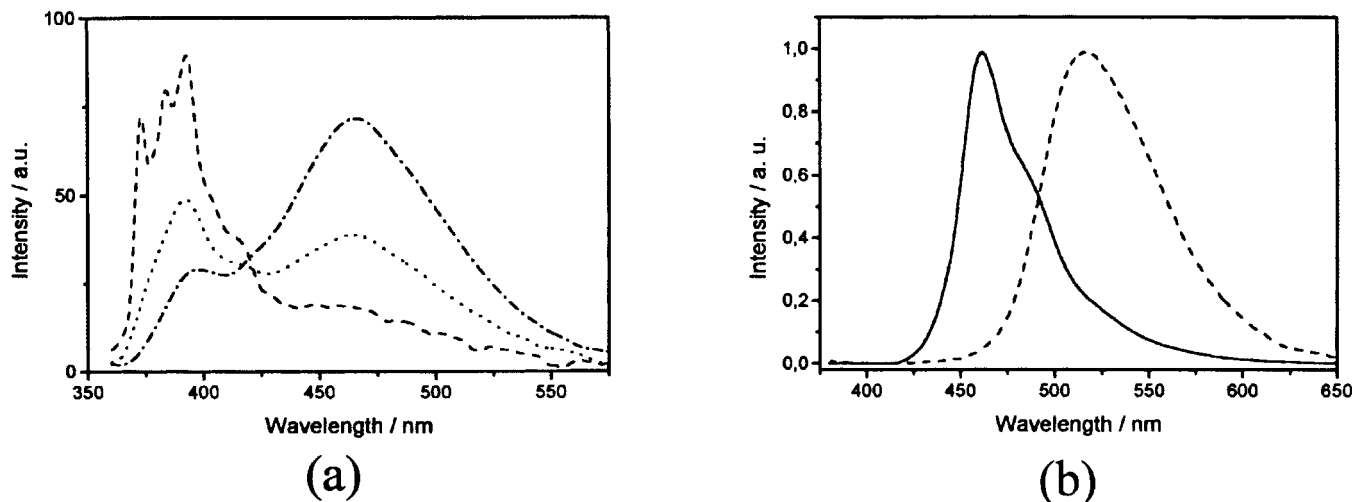
(a)   **(b)**

Figure 5.   Absorption spectra of dye solutions for different concentrations. (*a*) Pyrene in ethanol: (‑ ‑ ‑) 0.001 mol l^{-1}, ($\cdots$) 0.005 mol l^{-1}, and ($-\cdot-\cdot-$) 0.02 mol l^{-1}. (*b*) Tetrahexyl-pyrene: (——) 0.01 mol l^{-1} in chloroform and (‑ ‑ ‑) the pure compound.

3.3. *Electroluminescence*

The electroluminescent properties of the new compounds were tested by investigating thin layers of the substances between an ITO-coated glass substrate and an evaporated Al cover electrode. The most efficient electroluminescence was observed for the perylene compounds. For the compound **3a**, even a single organic layer between an ITO and an aluminium electrode shows a bright orange–red luminescence if the voltage exceeds a certain threshold value which depends on the film thickness (figure 6). The rise in the current starting at this threshold voltage coincides with the appearance of luminescence in the sample. In our first attempts, the threshold voltage was about 18–20 V. However, by optimizing the sample thickness *d*, it could be reduced to about 12 V (for $d \approx 50$ nm). The perylene compound is quite stable with respect to the ambient conditions.
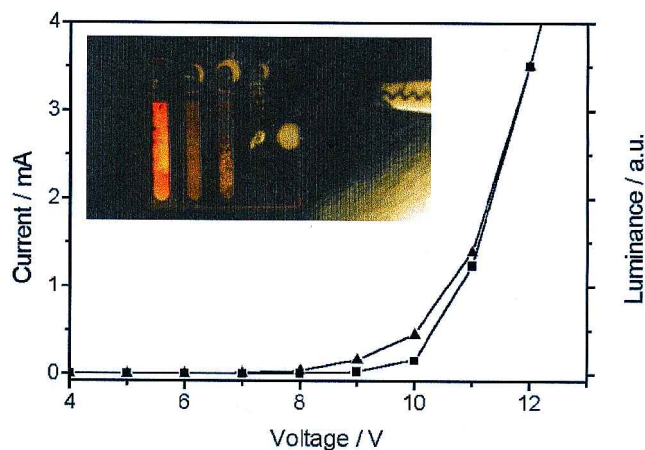

Figure 6.   (■) Current versus voltage and (▲) light intensity versus voltage for a single layer OLED containing the perylene compound **3a**.

Even when exposed to oxygen and moisture in the laboratory environment, the samples have lifetimes of typically about 20 h. However, voltages above 25 V lead to rapid degradation. For a single layer light emitting diode of the perylene compound **3a** exhibiting a threshold of 12–13 V, we measured a luminance of about 100 cd m^{-2} at 20 V. In the case of the perylene derivatives, our attempts to improve the performance by adding a hole transporting layer led to a spatially more uniform emission from the area covered with the aluminium electrode and to a greater stability, but did not result in a reduction of the threshold voltages. Images of the surface topography made by atomic force microscopy indicate that the additional layer has an important effect on the smoothness and the connectivity of the thin layers (figure 7). The ITO layer, figure 7(*a*), shows large spikes with a height in the same region as the total thickness of the organic layers. However, the surface of a TPD layer deposited on the ITO substrate by spin-coating is rather flat, figure 7(*b*), thereby providing a good substrate for the emitting layer, figure 7(*c*). The latter shows some platelets at the surface, but is also rather smooth.

The spectra of electroluminescence (figure 8) coincide with the spectra of photoluminescence (figure 2). Thus, blue and yellow–green electroluminescence can be achieved from the derivatives of triphenylene and pyrene, respectively. The side chains have a small influence on the luminescence wavelength. Thus, variations of the side chain do not change the emission dramatically, but allow for some fine-tuning of the colour. In contrast to the behaviour of the perylene derivatives, the use of a second organic layer was necessary to obtain electroluminescence from the compounds **1a**–**2i**. The occurrence of electroluminescence in liquid crystalline triphenylene derivatives was reported earlier by Wendorff *et al.*
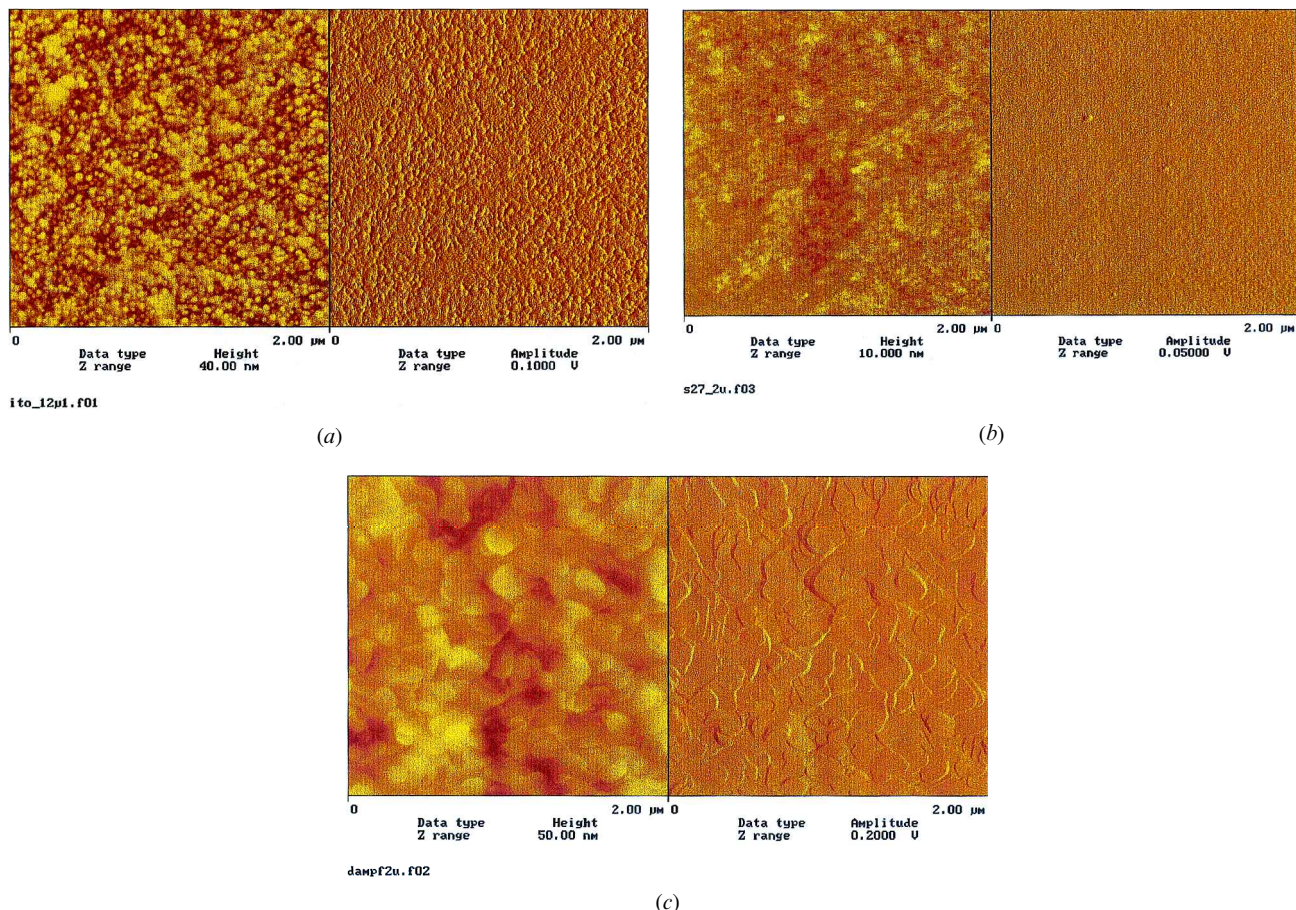
Figure 7. Topography of the surfaces, measured using atomic force microscopy. (*a*) ITO substrate, (*b*) TPD layer deposited by spin-coating on the ITO substrate, (*c*) perylene compound **3a** ($\approx$ 50 nm) deposited by thermal evaporation in high vacuum.
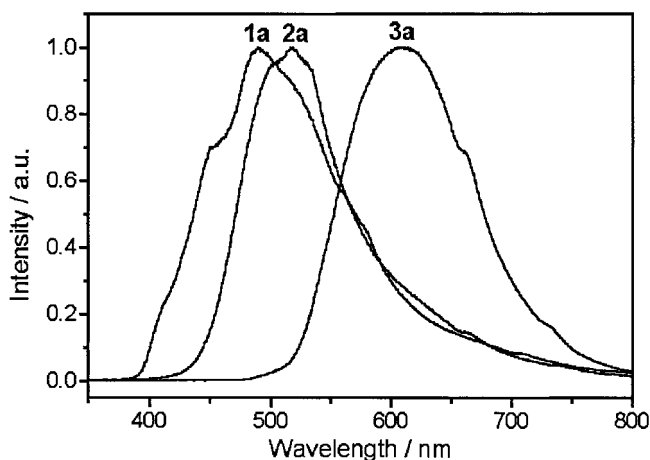

Figure 8. Electroluminescence spectra of the compounds **1a**, **2a**, and **3a**.

[23, 24]. However, they used hexa-ethers of triphenylene which are very effective hole conducting materials. In contrast, we expect our compounds **1a**–**1i** to have electron conducting properties because the ester groups

tend to reduce the electron density in the aromatic core. Consequently, their combination with a hole conducting layer of TPD proved to be useful. The threshold voltage of a two-layer device containing a triphenylene derivative was found to be about 18 V (figure 9), and a blue–violet electroluminescence is observed. Two-layer devices which were prepared under the same conditions, but contained poly-3,4-ethylene dioxythiophene as the hole transporting layer and a pyrene derivative (either **2a** or **2c**) as emitting layer, showed a lower threshold voltage, 5–6 V at $d \approx 50$ nm (figure 9). They show a yellowish green luminescence. Unfortunately, their intensity reached saturation at about 15 V, and showed a rapid decrease, probably due to degradation in the presence of air. Because of the rapid destruction, a precise value of the luminance could not be measured.

4. Conclusion

We have investigated the spectral characteristics of some new liquid crystals which are very promising for use in electroluminescent devices. The absorption spectra
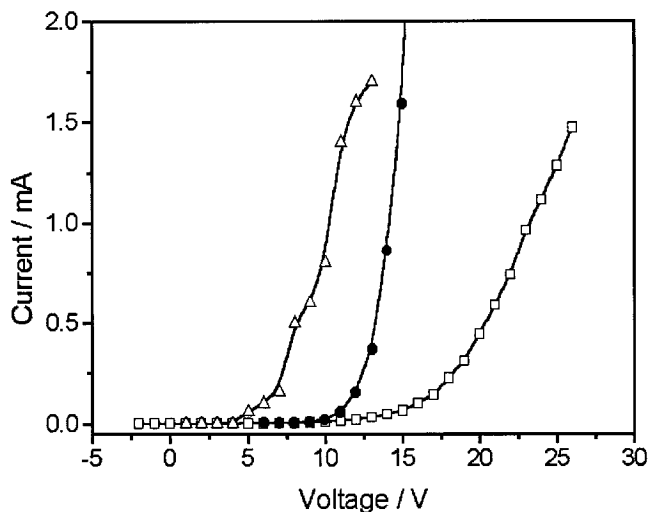
Figure 9. Current versus voltage for (□) a two layer OLED containing the triphenylene derivative **1a**, (△) a two layer OLED containing the pyrene derivative **2a**, and (●) a single layer OLED containing the perlyene compound **3a**.

of these compounds are in good agreement with calculations using the time-dependent density functional tight binding theory. The luminescence spectra cover different areas over the whole range of visible light. Comparison between the luminescence of the pure compounds and their respective solutions in organic solvents reveals a large spectral shift occurring for the pyrene and perylene derivatives, indicating the formation of excimers at sufficiently high concentrations. Preliminary studies on single and double layers of the compounds indicate that the new liquid crystals are promising candidates to be studied for electroluminescent devices. This first characterization of single and double layers is far from optimum in the conditions relating to charge injection, transport and recombination. Thus, we trust that much better performances can be obtained using the compounds presented.

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