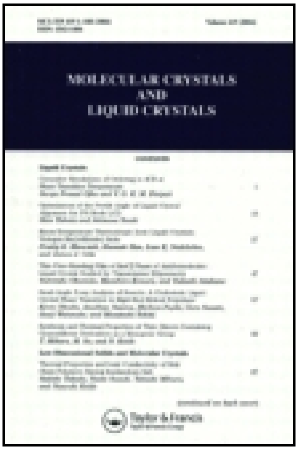

Molecular Crystals and Liquid Crystals

Low Molar Mass and Polymer Discotics: Structure, Dynamics and Opto-Electronic Properties

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LOW MOLAR MASS AND POLYMER DISCOTICS: STRUCTURE, DYNAMICS AND OPTO-ELECTRONIC PROPERTIES

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Columnar discotics display along the columnar axis unusually large mobilities for charge carriers. This fact can be exploited for instance in the areas of photoconductors and organic light emitting diodes. The transport takes place via hopping between localized transport sites. The theoretical analysis based on an analytical approach as well and on simulations predicts that the energetic distribution of the transport sites controls the particular dependence of the charge carrier mobility both on the temperature and the applied electric field. Narrow distributions cause the mobilities to increase and to become independent of temperature and field. A further prediction is that the mobility depends on the length of the columns.

We have varied the chemical structure of asymmetrically substituted triphenylenes with the purpose to enhance the spatial order and consequently to reduce the width of the energy distribution of the density of states. Novel methods of macroscopical orientations of the columns were developed. The highly ordered plastic columnar state achieved in this way was found to display a high one dimensional mobility and no field and temperature dependence of the mobility as obvious from Time of Flight experiments. The order within the columnar phase as well as the columnar length was furthermore modified by subjecting the discotics to geometric confinements with dimensions down to a few nm. Confinements imposed in PDLC systems and porous glasses give rise to strong changes not only in spatial structure and molecular dynamics and in the absorption and emission properties but also in LED-properties. Finally we have modified the optical properties of semiconductor quantum well structures by discotic layers.

Keywords: discotics; structure; orientation; glass transition; charge carrier mobility; light emitting diodes; hybrid structures

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INTRODUCTION

In 1888 F. Reinitzer reported a remarkable discovery [1]. He observed a substance that did not melt directly from the solid crystalline state to the isotropic fluid state but rather displayed an intermediate fluid state. In the following years it became obvious that this is a rather general phenomena, that many intermediate phases exist and that a large number of substances display such a behavior provided that the molecules have an elongated shape, i.e. are to a first approximation rod-like. The intermediate phases were called liquid crystalline phases since they were found to combine features characteristic of the crystalline but also of the liquid state. They possess unique optical, dielectric and elastic properties which eventually resulted in a broad range of applications, in particular in displays.

It took nearly one century before S. Chandrasekhar reported in 1977 that not only rod-like but also disc-like molecules display liquid crystalline phases–discotic phases [2]. The unique feature of the discotic phases is the spontaneous formation of one-dimensional stacks–columns. These columns, in turn, organize frequently on a two-dimensional lattice assuming a hexagonal or rectangular packing, for instance. Today a large variety of substances are known which display discotic phases, both low molar mass and polymer discotics exist [3]. The disc-like units may be incorporated in the latter case into the chain backbone or attached as sidegroups to it. Doped discotics, crosslinked discotics, blockcopolymers containing segments with disc-like building blocks, combinations of disc-like and rod-like units–the variety of discotic systems is overwhelming.

Most of the research activities following the discovery by S. Chandrasekhar were devoted to the synthesis of novel discotic materials, the evaluation of their thermal properties and of the arrangements of the discs in space. Yet slowly the insight emerged that discotics may have a bright future in applications due to the particular arrangement of the discs in space and the opto-electronic properties originating from them [3–8].

A key feature is the formation of one-dimensional stacks, of columns. The formation of the columns may give rise, for instance, to a significant blue shift of the absorption maximum, a shift which has been attributed to strong interactions between the excited disc-shaped molecules along the columns. Using quantum chemical calculations Markovitsi et al. were able to correlate the magnitude of the blue shift with the number of molecules within the columns interacting with each other [9,10]. The strong interactions between the discs along the columns give rise to particular transport properties for charge carriers and for optical excitations.

Applications as photo conductors in copy machines or laser printers, in organic light emitting diodes or in field effect transistors have been and are being considered. Discotics are of potential interest furthermore for optical storage, including holographic optical storage [11]. More recently they experience strong activities towards application as compensation layers in liquid crystalline displays.

DISCOTICS AND ELECTRONIC TRANSPORT PROCESSES

Optoelectronic properties of organic materials such as absorption, photo- and electroluminscence as well as the transport of charge carriers and optical excitations are known to depend strongly on the molecular state of order [12,13]. The spatial disorder characteristic of amorphous materials is known to give rise, for instance, to localized transport states the energy and distance of which are subjected to a distribution. Assuming the presence of such distributions Bäßler et al. were able to account for the experimentally observed temperature and field dependence of charge carrier mobilities in organic amorphous materials including amorphous polymers and guest host systems [13–17].

The transition from the short range order characteristic of perfectly amorphous materials to the more regular though noncrystalline order displayed by liquid crystalline materials has been found to lead to significant changes both in electronic transport processes as well as in excitation and emission properties. It has been furthermore reported, in particular for triphenylene derivatives, that the formation of a hexagonal columnar phase from the isotropic melt leads to an increase of the hole mobility by at least two orders of magnitude [4]. Columnar discotics have therefore met with increasing interest for applications in areas such as light emitting diodes [6,7,18,19] or photoconductors. It might be important for such applications to be able to control their optoelectronic properties and transport properties in a broad range. One expectation is that both excitation and emission properties but also electronic transport properties should depend strongly on the perfection of the liquid crystalline order and on the magnitude of the correlation length along the columns and perpendicular to them. X-ray studies have revealed that the correlation length along the columns within the bulk state amounts typically to about 15 to 20 nm as far as triphenylene systems are concerned and that these values do not vary strongly with temperature [7,20].

The transport takes place via hopping between localized transport sites (Fig. 1). The theoretical analysis based on an analytical approach as well and on simulations predicts that the energetic distribution of the transport sites controls the particular dependence of the charge carrier mobility both on the temperature and the applied electric field [21,22]. Narrow distributions cause the mobilities to increase and to become independent of

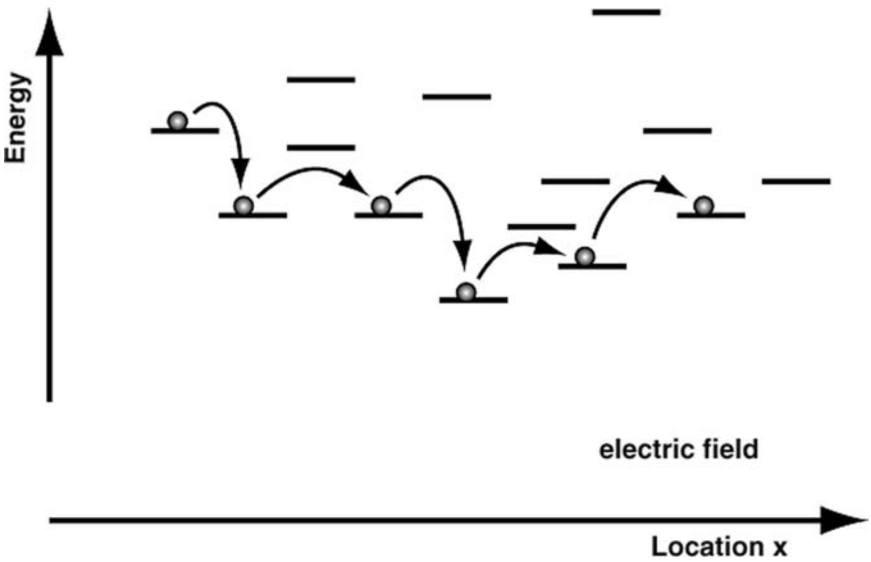


FIGURE 1 Density of states of localized transport sites.

temperature and field. A further prediction is that the mobility depends on the length of the columns. This is apparent from Figure 2 displaying the dependence of the mobility on a parameter containing the temperature and the width σ of the energy distribution. The assumption based on these results is that the transition from the hexagonal to a more highly ordered columnar state should increase the mobility even more and should make it independent of temperature and electric field.

Our interest since many years is directed at tuning opto-electronic properties of discotics via modifications of their chemical structure and arrangement in space and at using discotic systems as photoconductors, in light emitting diodes and for optical storage. A brief survey will be given below on the main results of these investigations.

COLUMNAR DISCOTICS WITH ENHANCED ORDER [23,24]

Columnar discotic phases are ideal model systems for studying the correlation between order and optoelectronic properties, in particular transport processes. They cover the range between real crystals and disordered amorphous systems nearly continuously [3]. Planar nearly disc-like molecules form columns with a regular or irregular placing of the molecules in the columns (ordered: o; disordered: d). The columns

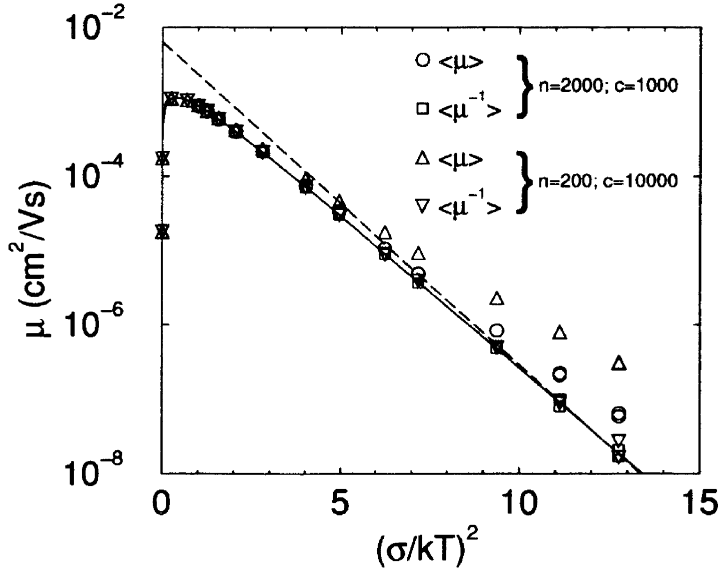


FIGURE 2 Dependence of mobility on temperature and widths of the density of states.

either display just an orientational order or form a two dimensional lattice.

More recently we have discovered for asymmetrically substituted triphenylenes a novel discotic columnar state characterized by a three-dimensional crystal-like registry and an orientational disorder of the molecules about the columnar axis [23,24]. The enhanced order is obvious from the X-ray diagram (Fig. 3) showing a doublet rather than a singlet reflection at larger scattering angles. In addition mixed reflections (hkl) were observed. Typical triphenylene derivatives used for the investigations are shown in Scheme I.

ASYMMETRICALLY SUBSTITUTED TRIPHENYLENES

For selective systems we observed the occurrence of a reentrant phase behavior involving the highly ordered discotic phase [25].

Some triphenylene derivatives displayed, in addition, a small angle reflection indicative of a supramolecular structure. The superstructure arises from a specifc orientational order of the lateral substitutens, as shown schematically in Figure 4.

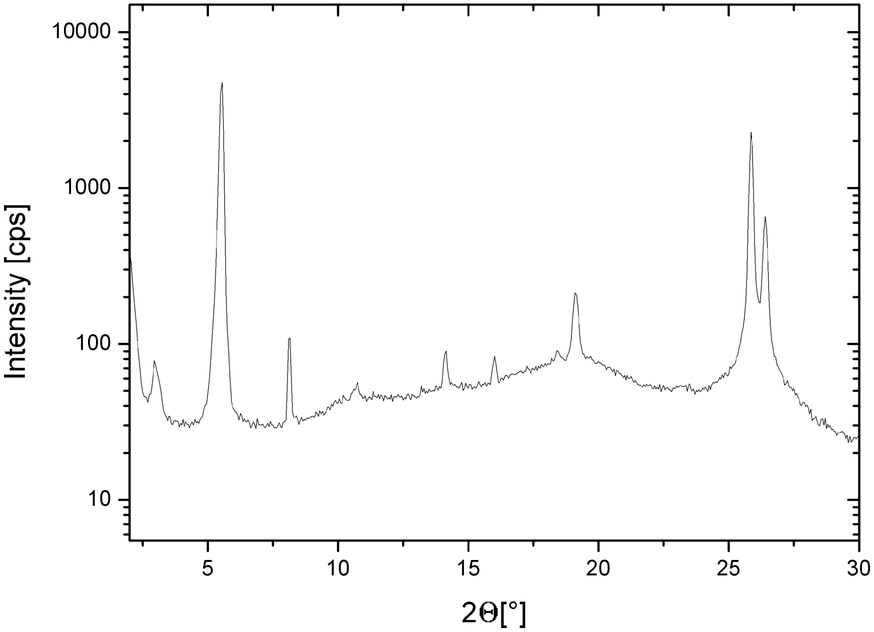
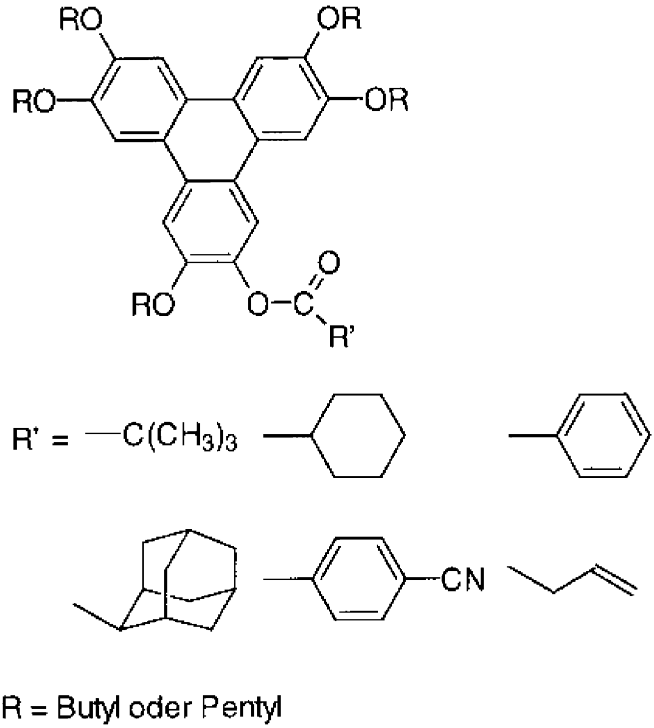


FIGURE 3 X-ray diagram of the plastic columnar phase.

GLASS FORMATION [26]

The applications introduced above require, in general, that solid glassy films can be obtained from the discotic materials retaining the particular arrangements of the molecules characteristic for the discotic phases. The transformation of a supercooled fluid state into a solid glassy material plays an important role in material science [27–29]. Transport properties, thermodynamic and mechanical properties but also electric and optical properties are known to change significantly as the glass transition process takes place. It is for this reason that strong activities have been devoted towards developing a detailed understanding of the freezing-in process and the nature of the glassy state, using both experimental and theoretical approaches [30–33]. Most experimental investigations were concerned in the past with disordered materials i.e. with the freezing-in of the supercooled melt. A small number of investigations have focussed on supercooled plastic crystals characterized by the presence of a threedimensional translational symmetry and rotational disorder [34,35].

We have compared the character of the glassy state of highly ordered discotic glasses with the one of disordered glasses. One promising



SCHEME 1 Asymmetrically substituted triphenylenes.

approach is based on the analysis of the fragility. Angell was the first to point out that glasses may be classified with respect to the dependence of the viscosity–or the relaxation time, for that matter–on the temperature in

FIGURE 4 Discotics with supramolecular structure arising from a mutual orientation of the lateral substituents. (See Color Plate I).

the neighborhood of the glass transition temperature [36]. Strong glasses display an Arrhenius type of relationship

$$\tau \propto \exp(E_A/T)$$

and fragile glasses a relationship which can be represented by a WLF-equation or a Vogel Fulcher type of equation [37,38]

$$-\log\frac{\tau_T}{\tau_{T_g}} = \log\frac{\nu_T}{\nu_{T_g}} = \frac{C_1(T - T_g)}{C_2 + T - T_g}$$

or

$$\tau \propto \exp(A/T - T_o)$$

The finding is that organic glasses, van der Waals glasses, both of low molar mass and polymer origin, tend to be fragile whereas inorganic glasses such as SiO_2 glasses tend to be strong glasses.

The character of the glass transition can be studied by dielectric relaxation investigations. Figure 5 displays as an example the dependence of the dielectric loss on the temperature for various frequencies. Considered is the case of the para cyanobenzoate triphenylene which displays a hexagonal ordered phase.

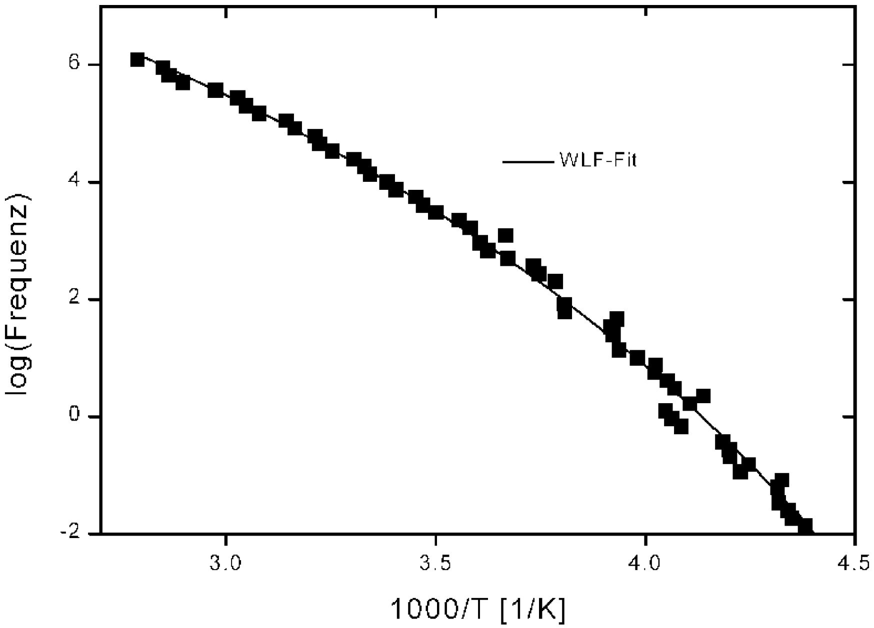


FIGURE 5 Activation diagram for the dielectric relaxation of the para-cyanobenzoate triphenylene displaying a hexagonal phase.

The observation is that a strong relaxation takes place, which can be assigned to the glass relaxation. This assignment is based also on calorimetric investigations. The relaxation frequencies–temperature dependence is displayed in Figure 5. It is apparent that this dependence can be represented by the WLF- equation (see fit). The WLF parameters obtained from this plot are well within the range reported for disordered glasses [37,38]. The extrapolation of the plot to a low frequency value of 1/100 Hz yields a glass transition temperature, which agrees with the one obtained from calorimetric studies. All hexagonal phases studied so far show a WLF-behavior with WLF constants within the same range: the discotic hexagonal ordered phase behaves as a fragile glass in all cases.

A totally different result is obtained for the plastic columnar phase. Figure 6 displays the activation diagram obtained for the glass relaxation of a particular compound.

It is obvious that the relaxation frequency dependence obeys an Arrhenius law with a constant activation energy, which amounts to 92 kJ/mol in this particular case. All plastic columnar phases seem to display an Arrhenius behavior and all activation energies are very similar of the order of 90 kJ/mol. The experimental finding thus is that ordered discotics

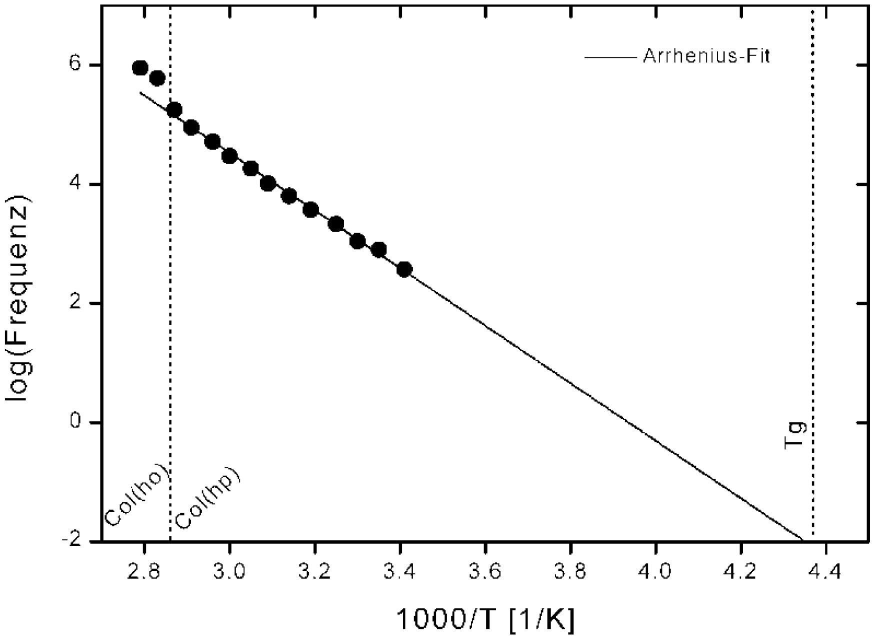


FIGURE 6 Activation diagram for the dielectric relaxation of the triphenylene displaying a plastic columnar phase.

can either be classified as strong or as weak glasses depending on the structure of the columnar phase independent of the chemical structure of the discotic compounds. The highly ordered plastic columnar state behaves as a strong glass former whereas the hexagonal ordered phase behaves as a fragile glass as far as the dependence of the relaxation time on the temperature is concerned. The magnitude of the activation energy and of the WLF coefficients respectively are similar in all cases studied here.

MACROSCOPIC ORIENTATION [39,40]

The requirement for applications of discotics in light emitting diodes or field effect transistors is that films with a uniform orientation of the columns can be achieved. The orientation should be along a given direction in the plane of the film as far as field effect transistors are concerned and along the film normal as far as light emitting diodes are concerned.

Spin coating tends to lead to a planar orientation of the columns with no preferred orientation within the plane. The corresponding X-ray reflection studies reveal just the presence of the hexagonal reflection as shown in Figure 7. A polytetrafluoroethylene orientation layer deposited along a given direction in the plane leads to a preferred orientation of the columns within the plane of the film along the direction of deposition [40]. This is apparent from dichroic studies displayed in Figure 8. Finally annealing of the films at temperatures close to the isotropization temperature lead to a reorientation of the columns along the film normal. Figure 7 shows the corresponding X-ray diagram displaying now just the intracolumnar reflection. The disadvantage is that the reorientation tends to be accompanied by a partial dewetting leading to the formation of a hexagonal pattern in many instances.

MOBILITY OF CHARGE CARRIERS [41,42]

The mobility of charges is in general investigated by time of flight experiments. The transition from the isotropic phase to a columnar phase manifests itself in general by an increase of the mobility by at least two orders of magnitude. The more highly ordered plastic columnar phase displays a mobility which is higher by another one or two orders of magnitude as expected form the theoretical analysis discussed above. Furthermore we observe that the mobility is independent from both the temperature and the electric field [41].

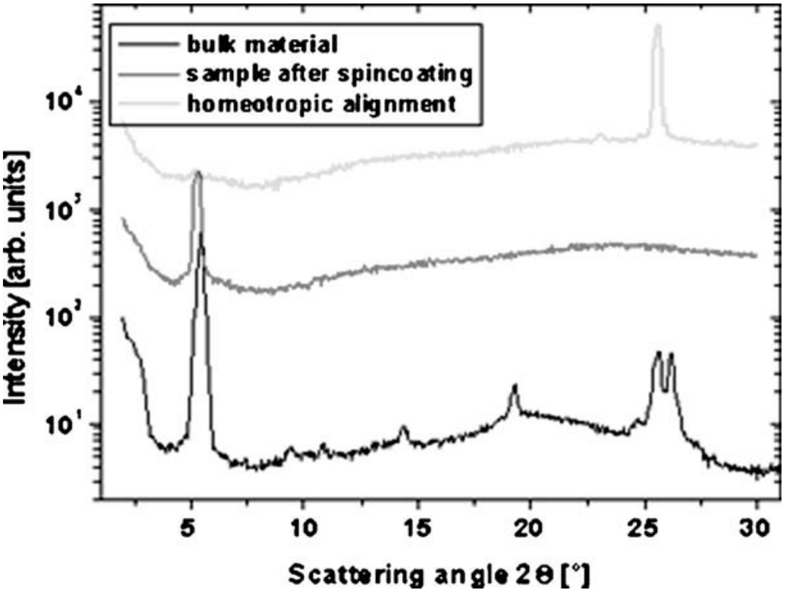


FIGURE 7 X-ray diagram characterizing different types of orientation with respect to the plane of the substrate. (See Color Plate II).

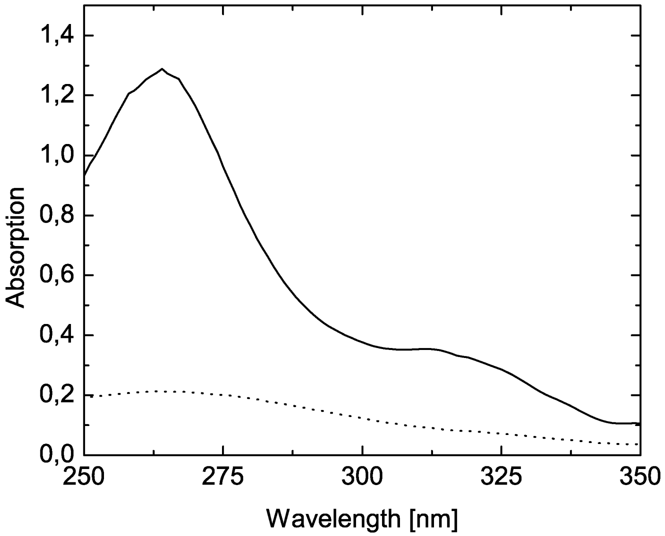
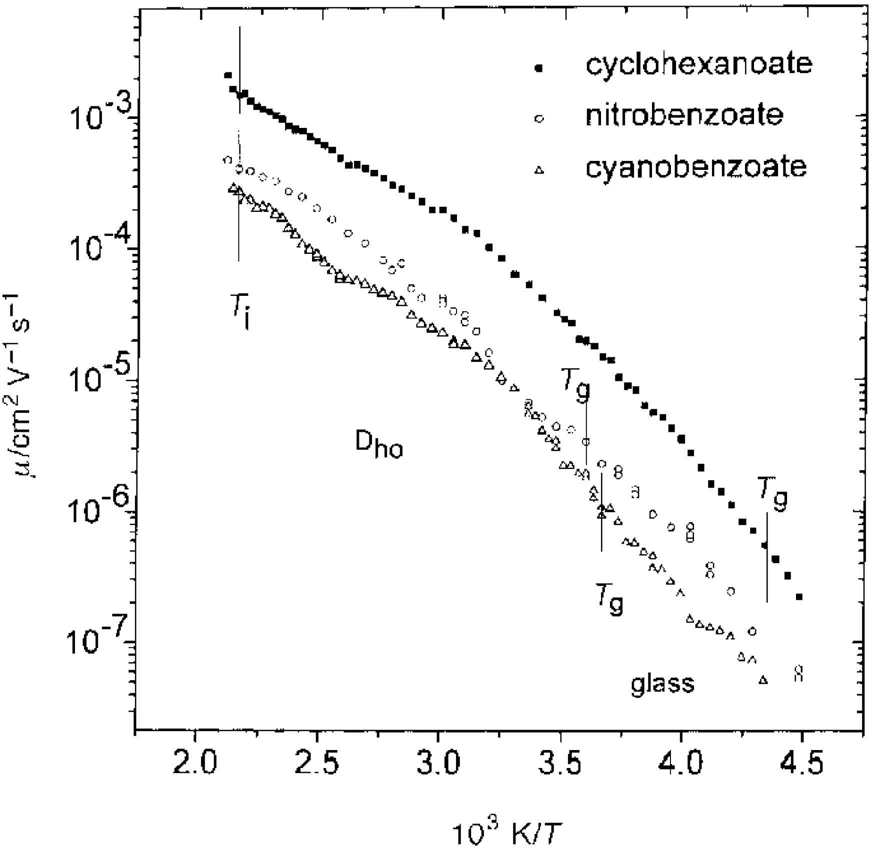


FIGURE 8 Dichroic studies on the orientation of columns on a PTFE layer.

To test the predictions of the theory we have introduced strong dipole moments into the triphenylenes [42]. The expectation is that these give rise to enhanced fluctuations of the local potential which in turn will increase the width of the energy distributions of the density of states. In fact the experimental finding is that the presence of dipoles reduces the mobility by one order of magnitude at a constant perfection of the spatial order. In addition we find that the mobility becomes dependent both on the temperature and on the field according to the predictions of the theory. This is shown in Figure 9.



(a)

FIGURE 9 Dependence of mobility on temperature (a) and electric field (b) for the dipole substituted triphenylene compound.

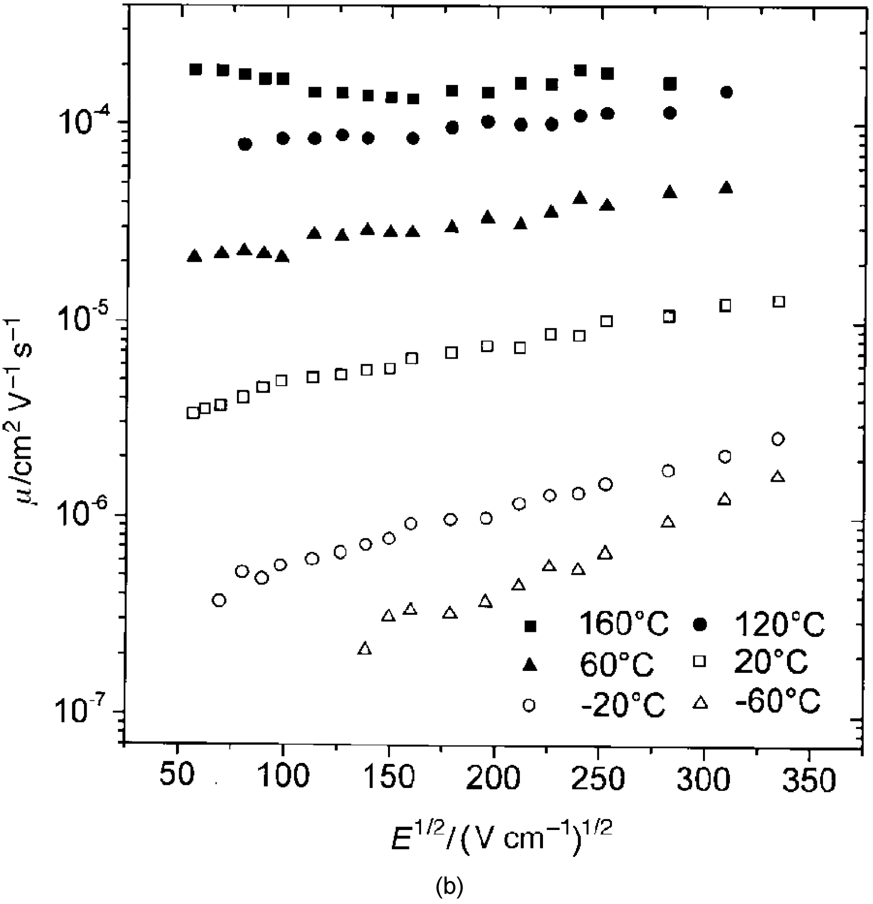


FIGURE 9 Continued.

LED APPLICATIONS [6,7,18,19]

Discotics, both low molar mass and polymeric systems, were incorporated as functional films in light emitting diodes sandwiched between the electrodes. Triphenylenes act in general, as good hole conducting layers yet discotics can be chemically modified to serve also as electron conducting layer. Chromophores were either solved within the discotic films or were located in separate layers. In the following results will be presented on the triphenyle systems introduced above.

Figure 10 shows the diode characteristic, i.e. the dependence of the current density on the applied electric field or voltage. The general

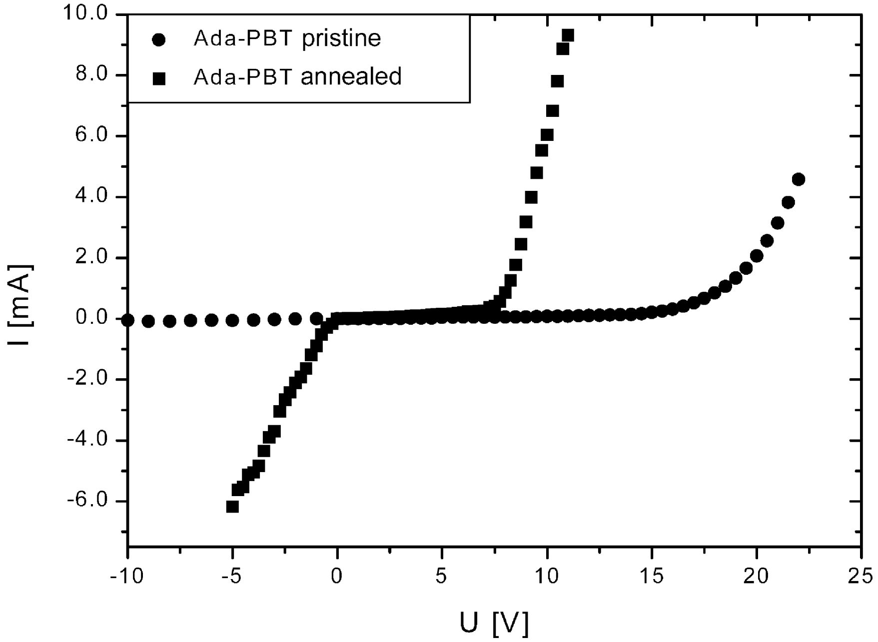


FIGURE 10 Diode characteristic of an LED composed of a discotic layer: as obtained by spincoating and after annealing at elevated temperatures.

observation is that the current sets in above a characteristic threshold field. Displayed are in Figure 10 the diode characteristics for single layer diodes directly after spincoating where a planar orientation of the columns is achieved and after annealing leading to a reorientation of the columns. The reorientation causes a significant reduction of the threshold field.

Similar effects were observed for a main chain polymer discotic, as apparent from Figure 11. Spincoating leads to an amorphous state, short annealing to the formation of columns more or less randomly oriented within the film and finally prolonged annealing cause a preferential orientation along the film normal. The threshold field is reduced for each of the steps described above.

Multilayer discotics, obtained for instance via Langmuir Blodgett techniques and discotic films obtained by vapor deposition have been reported in the literature as well as a very high luminescence of the order of more than 10000 cd/m^2 [8].

Pure discotics show electroluminescence, which resembles the fluorescence spectra closely (Figure 12), yet as pointed out above, in general the emission is controlled by chromophores mixed with the transport layer or located in separate emission layers.

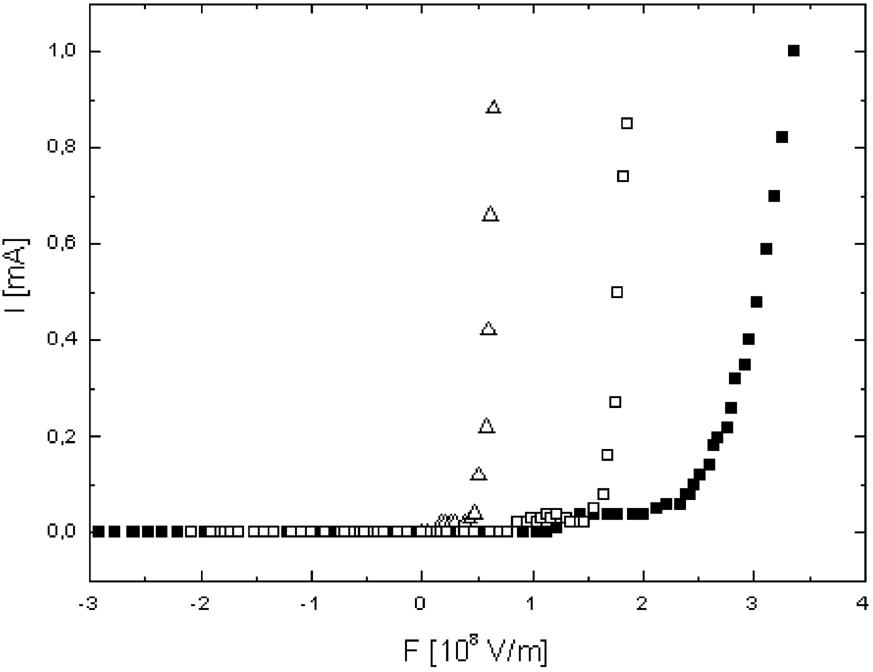


FIGURE 11 Diode characteristic of an LED composed of a discotic layer (main chain polymer); as obtained by spincoating; after annealing at elevated temperatures for five minutes, for one hour.

CONFINEMENT EFFECTS [43,44]

One effective way to control the structure of columnar discotics including the correlation length at a fixed temperature consists in subjecting the discotic material to geometric confinement effects. The influence of geometric confinements on the state of order and on the molecular dynamics of liquid crystals has been the subject of a considerable number of investigations [45–55]. The findings based on theoretical considerations and experimental results are that thermodynamic, structural and dynamic properties are strongly modified due to confinement effects. In fact, we were recently able to show that structure and molecular dynamics of columnar discotics can be strongly varied by introducing the materials into narrow pores provided by porous glasses [43]. Yet for technical application involving thin films the use of porous glasses meets with considerable difficulties.

The route which has recently been exploited by us is based on a fine dispersion of the discotic materials in a solid transparent polymer matrix.

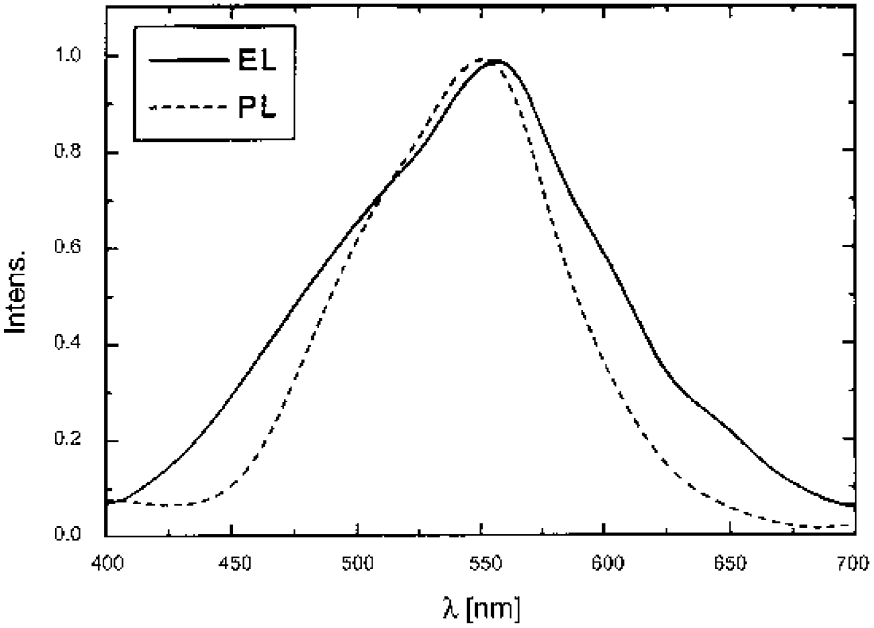


FIGURE 12 Fluorescence and electroluminscence of a LED with a discotic layer.

Solutions of the triphenylene compounds and the amorphous polymers poly(methylmethacrylate) (PMMA) or poly(carbonate) in chloroform as solvent were used for spincoating thin films. Depending on the spincoating conditions, the concentration of the discotic material relative to the polymer matrix and subsequent annealing conditions one is able to control the degree of dispersion of the discotic materials in a broad range extending from a molecular dispersion all the way up to phase separated structures in the μm range with corresponding variations of structural, dynamic and–most important–optoelectronic properties.

Using optical microscopy and AFM-studies it became apparent that the transparent films with a thickness of some μm are characterized by very small dispersed discotic regions having the shape of cylinders. The diameter was typically in the range of 200 up to 300 nm and the length of the same order of magnitude. No such structures were apparent in the 100 nm thick films indicating possibly a molecular dispersion or a dispersion with dimensions well below a few 100 nm

The X-ray diagrams of the confined and the bulk materials differ in the range of larger scattering angles. Here we observe for the bulk material a doublet reflection superimposed on the amorphous halo. The doublet is a signature of the plastic columnar state.

For the confined material we observe in the same temperature range a rather broad reflection resulting from a regular packing of the disc-like molecules along the columns for the bulk material as found for the hexagonal ordered state ($\mathrm{Col}_{\mathrm{ho}}$). It is thus apparent that the confinement tends to cause a destruction of the plastic columnar phase ($\mathrm{Col}_{\mathrm{hp}}$) and the induction of the less ordered $\mathrm{Col}_{\mathrm{ho}}$-phase. The dispersion of the discotic material in a solid polymer leads thus to significant modifications of the columnar structure: the plastic columnar phase may be destroyed and thus the three-dimensional registry.

The bulk material has been found to correspond in the plastic columnar state to a strong glass with a thermally activated relaxation behavior rather than to display a WLF-behavior (fragile glass). The confinement destroys the plastic columnar phase (see X-ray results above) and induces a hexagonal columnar phase. Such a phase, in turn, is known to correspond to a fragile glass.

The UV-Vis maximum of the bulk material considered here is characterized by a blue shift compared with the one found for solutions. A blue shift of this kind has been attributed to strong exciton interactions leading to collective excited states along the columns as apparent from quantum chemical calculations [9,10]. According to these calculations performed on symmetrical triphenylenes the magnitude of the shift depends on the state

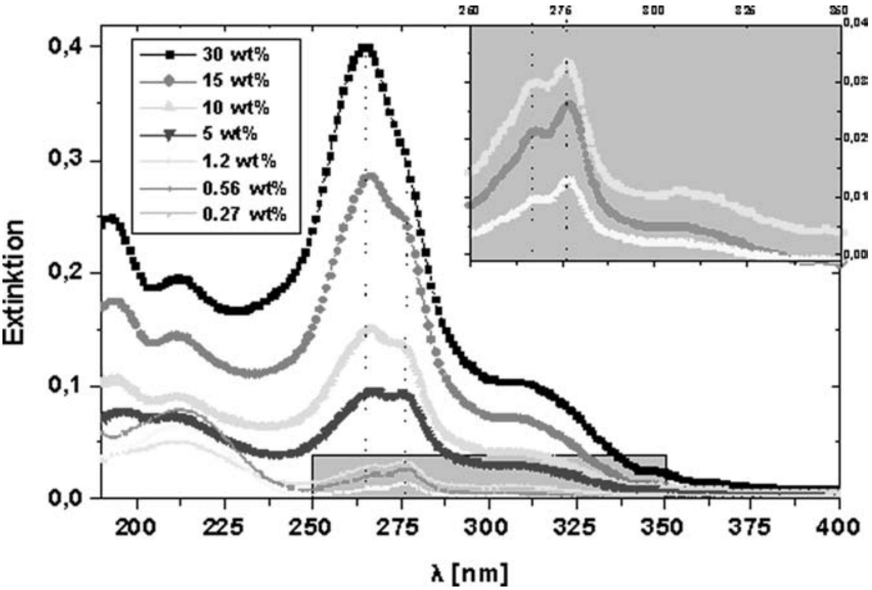


FIGURE 13 Absorption spectra for polymer dispersed discotics for various concentrations of the discotic. (See Color Plate III).

of organization in particular on the number of excited molecules interacting within a column.

The spectra obtained for films with lower concentrations of the discotics are very similar to the ones found for solutions indicating a fine dispersion approaching even a molecular dispersion. With increasing concentration there are two significant changes: firstly it is obvious that there is an exchange of the relative intensities of the two major vibronic bands (attributed to $v_4 = 0$ and $v_4 = 1$) of the $S_0 \rightarrow S_4$ transition and secondly a slight blue shift of both vibronic bands occurs (Figure 13). The final state found for the larger aggregates is an absorption behavior resembling the one of the bulk material where the $v_4 = 0$ vibronic band is too weak to be visible. These changes are apparently related to the increasing aggregate sizes of the dispersed material at higher concentrations. We tentatively attributed the changes in the intensity of the vibronic bands to slight changes in the anharmonic potentials of the ground and excited states such as slight relative displacements. The observed behavior is not in full agreement with the expectations based on the model calculations discussed above.

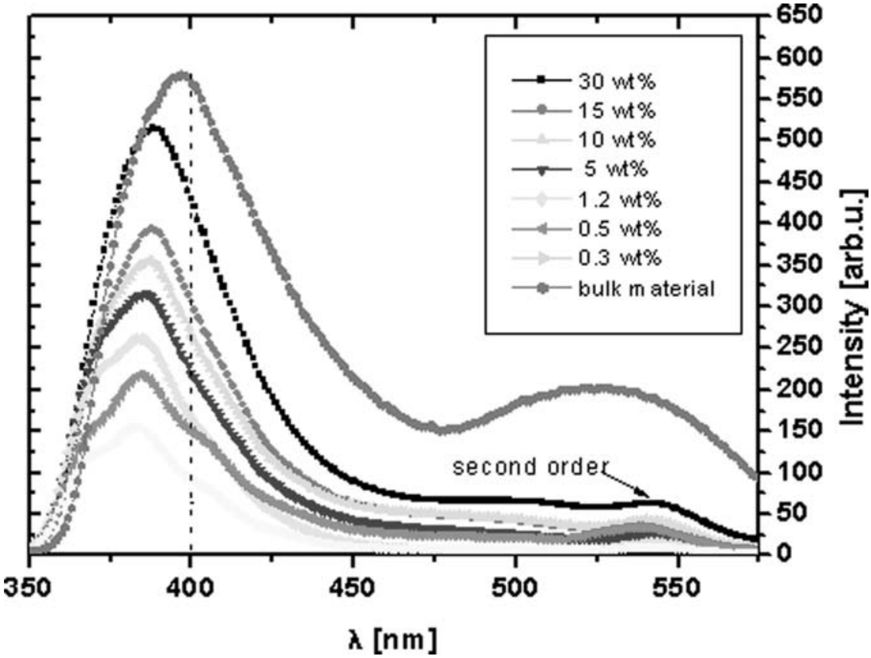


FIGURE 14 Fluorescence spectra for polymer dispersed discotics for various concentrations of the discotic. (See Color Plate IV).

For bulk materials in the plastic columnar phase we find that the fluorescence is red-shifted compared to the one found in solution the shift amounting to about 10 nm (Figure 14) from 385 to 395 nm. The dispersion of the discotic material in the polymer film causes a blue-shift of the emission maxima relative to the bulk value the spectra approaching thus the ones found for solutions. The emission spectrum is characterized in this case by a fine structure apparently related to vibronic contribution. Such a fine structure has also been observed for solutions. This may be taken as an indication for the presence of a molecular dispersion with the polymer acting as a solid solvent. Yet we observe similar spectra for the phase separated systems. An increase of the size of the dispersion causes a reduction of the fine structure of the emission band but no shift of the location of the emission band i.e. the emission band remains located at 385 nm. The location of the band is thus a function of the phase structure (Col_{hp}) and the fine structure a function of the size of the aggregate.

Finally we will consider the diode characteristics of the dispersed materials compared to that of bulk discotic material and pure matrix polymer which are displayed in Fig. 15. It is obvious that the pure matrix

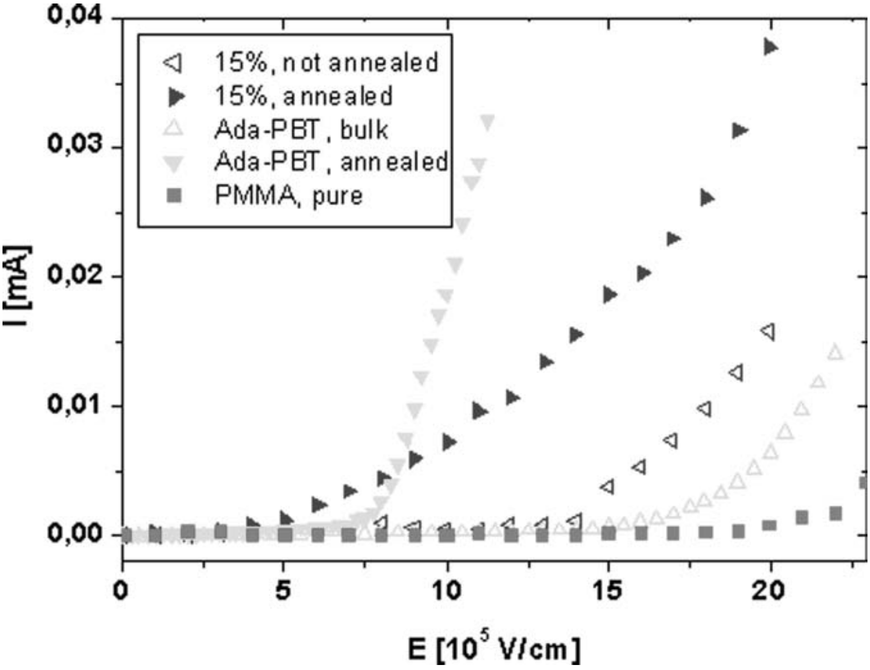


FIGURE 15 Diode characteristic for polymer dispersed discotics for various concentrations of the discotic compared with the bulk behavior. (See Color Plate V).

polymer does not contribute to the injection and charge carrier transport in any significant way and that the shape of the curves as well as the onset voltages differ from the ones of the bulk discotic material in LEDs. The onset voltage is rather high for the finely dispersed state. Yet, it decreases strongly after annealing the device at temperatures well above the glass transition temperature of the matrix polymer. This may be interpreted in terms of the formation of larger columnar discotic assemblies, which are responsible for an enhanced charge carrier transport. In fact, after annealing we observe cylindrical structures with the length of several hundred nm which is obvious from AFM-studies. These findings stress again the important role of the columnar state on injection and transport properties.

The dispersion of the columnar discotic material in a solid polymer matrix thus causes significant changes in structural and dynamic properties of the discotic material. Such a dispersion can be used to modify absorption and emission properties of the discotic material due to geometric confinement effects. Thin films, which are optically transparent, can be obtained via spincoating. These are of interest in optical applications such as organic light emitting diodes.

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