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Polarized Photoluminescence and Measurement of Degree of Order in Mixed Oriented Polymer Liquid Crystal Films

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Optical anisotropy in a new form of cross-linked liquid crystal network incorporating a fluorescent chromophore has been demonstrated. Linearly polarized absorption and fluorescence is observed in oriented thin films. From measurement of absorption and fluorescence anisotropy, the order parameter was found to be *0.65* and 0.80 respectively. Temperature dependent birefringence measurements reveal that there is no appreciable loss of alignment up to 200°C.

Raman spectroscopy was used to investigate the discrepancy between the absorption and fluorescence measurements. It is found that the fluorescent chromophore is less well ordered than the liquid crystal. The reasons for this are discussed.

Keywords: Photoluminescence optical anisotrophy; polarized absorption; fluorescence; order parameter; oriented films

INTRODUCTION

The last few years has seen many advances in the field of organic optoelectronics, specifically, light emitting devices using polymers and

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other smaller molecules [l]. The performance of such devices has improved considerably due to optimisation of material interfaces, improvement of quantum yield and a better understanding of charge transport processes in amorphous organic systems.

The point has now been reached where more sophisticated devices such as organic microcavity structures [2] and polarized emitters *[3]* are beginning to be reported. In any study of new systems, the starting point is characterisation of the optical (absorption and photoluminescent) properties. This paper therefore presents the optical characterisation of a new method of inducing polarized photoluminescence by the use of aligned, cross-linked liquid crystal networks.

To date, work in this area has employed a number of different alignment techniques designed to induce polarized emission. The first technique is controlled chemical vapour deposition **(C.V.D.)** which has been used to produce polycrystalline thin films of small conjugated organic molecules [4]. The second technique is Langmuir - Blodgett film deposition which was used to fabricate polarized polymer light emitting diodes *[5].* Finally, polarized luminescent films have been produced from functionalised polymer liquid crystals by substrate rubbing-induced alignment in the liquid crystal phase [6].

These techniques have been applied with varying degrees of success; there are still some problems to overcome. **All** of the techniques have so far failed to produce particularly high degrees of linear polarization.

The approach which we have used in an attempt to overcome some of these problems is *in-situ* photopolymerisation of functionalised oriented liquid crystals to produce highly aligned chromophore networks. This technique has been studied for a number of years now [7] for potential application in solid state liquid crystal display devices. **Its** exploitation in other areas is still however limited.

To produce these oriented polymer networks, *in-sicu* photopolymerization of macroscopically oriented mixtures of liquid crystal (LC) diacrylates was used. Figure 1 depicts the photopolymerisation process in the liquid crystal phase. The technique involves the macroscopic alignment of the LC diacrylates and the 'freezing-in" of the orientation by photo-polymerization. Previous work has invariably employed molecules where the saturated carbon chains are positioned at either end of the long axis of the liquid crystal core *[7].* In our case however, the alkyl chains are bonded across the short axis of the core (see Fig. 1).

FIGURE 1 Schematic of *in situ* photopolymerization.

Networks obtained by this method are highly cross-linked and well ordered. This high degree of orientation is both thermally and temporally stable.

RESULTS AND DISCUSSION

In this study, we sought to utilise the long range ordering characteristics of liquid crystals to produce a thin film which would preferentially emit light in a particular polarization and would exhibit good thermal stability properties. We therefore set out to design and synthesise a liquid crystal with an emissive functional core and cross-linkable side-groups. Our aim was to utilise such a liquid crystal in an ordered thin film form.

Synthesis and Characterization

In order to produce oriented films with linearly polarised emission characteristics, a chromophore with a blue/violet emissive core and acrylate functionalities **(111)** was synthesised. The synthetic route to **I11** is depicted in Scheme I. **A** similar molecule without any functionalities at the end of the alkyl tails was previously synthesised **[8]** and the same general method was used to prepare **11. I11** was characterized by IR, ¹H, ¹³C DEPT NMR and UV/visible spectroscopy.

Differential scanning calorimetry shows that **I11** exhibits a small enthalpy phase transition at **35°C** and a larger transition (to the isotropic phase) at 80°C on the heating cycle. On the cooling cycle however, no phase transition are observed. This is possibly due to slow recrystallization since on cooling overnight, the phase transitions return.

SCHEME 1 Synthesis route to compound **111.**

111 exhibits strong blue/violet fluorescence in dilute chloroform solutions. Figure 2 shows the electronic absorption and fluorescence spectra of the compound in dilute toluene solution. The quantum yield of fluorescence in chloroform solvent was determined by a comparative method [9] and is 60% in dilute solutions of cyclohexane.

Phase Transition Studies

The phase transitions of compound **111** were studied using differential scanning calorimetry **(DSC)** and polarizing microscopy. The material is crystalline at room temperature and on heating shows a phase transition of **38°C** to an unidenified intermediate phase. The transition from this intermediate phase to the isotropic phase has an onset temperature of 67°C and peak is at 80°C. The sample exhibits no phase transitions when it is cooled from the isotropic phase until below 30° C where it crystallizes.

FIGURE 2 Absorption (solid line) and fluorescence (dotted line) spectra of a toluene solution of **III**.

Long Range Ordering

A mixture of **III**, 2% mol. of photoinitiator (CIBA-IrgacureTM 651) and 0.1 mole percent of thermal inhibitor (4-methoxyphenol) was prepared.

A glass cell was filled with the mixture in molten form by capillary action. The sample was cooled overnight to allow recrystallization. The sample was heated to its intermediate state (50°C) and any ordering in the film was observed through cross-polarizers under a microscope. Unfortunately no long range ordering was observed in this intermediate phase.

In order to produce a low viscosity nematic phase, *5%* by weight of **111** was mixed with, 1,4-phenylene **bis{4-[6-(acryloyl-oxy)hexyloxy]** benzoate) **(C6H)**, a previously studied cross-linkable nematic liquid crystal [lo]. The same proportion of photoinitiator and thermal inhibitor as before was then added. This new mixture exhibits a crystalline-nematic phase transition at 116°C and a nematic to isotropic transition at 150°C. Glass cells containing this mixture were found to exhibit long range in the nematic phase. It is possible to 'freeze-in' this long range ordering by photopolymerization. This procedure was found to be effective for loadings of up to 15% by weight of **III**.

Polarized Absorption and Emission Studies

In order to measure optical anisotropy in the film, polarization dependent UV/visible absorption spectra were recorded. Spectra recorded for a polarization perpendicular and parallel to the director and normalised for the polarizer absorption are shown in Figure 3.

Clearly, the oscillator strength in absorption is concentrated along the direction of rubbing. The order parameter, **S** was calculated by comparing peak absorption (355 nm) parallel and perpendicular to the direction of rubbing according to Eq. (I).

$$
S = \frac{A_{\text{PA}} - A_{\text{PE}}}{A_{\text{PA}} + 2A_{\text{PE}}}
$$
 (1)

where A_{PA} and A_{PE} are the values of absorbance parallel and perpendicular to rubbing respectively. S for absorption was thus found to

FIGURE 3 Linear polarization dependence of optical absorption.

be 0.65 which compares favorably with values found for similar nonfluorescent systems [10]. It is noteworthy that there appears to be an extra absorption band in the perpendicular absorption spectrum. The origin of this band is not clear, it may perhaps be a weak vibrational mode (bending mode) polarized primarily across the long axis of the fluorescent molecule. Whatever the origin of the band, its presence reduces the value of **S** as determined by absorption.

The emission spectra (for an excitation wavelength of 340nm) parallel and perpendicular to the rubbing direction are shown in Figure 4. There is obviously a strong linear polarization of the blue/ violet emission. By way of comparison, the order parameter was again determined by comparing the numerically integrated emission spectra for the parallel and perpendicular directions according to a modified form of **Eq.** (1):

$$
S = \frac{\int_0^\infty E_{\text{PA}} d\lambda - \int_0^\infty E_{\text{PE}} d\lambda}{\int_0^\infty E_{\text{PA}} d\lambda + 2\left(\int_0^\infty E_{\text{PE}} d\lambda\right)}\tag{2}
$$

FIGURE **4** Linear polarization dependence of photoluminescence.

where E_{PA} and E_{PE} are the emission intensities parallel and perpendicular to orientation respectively.

In this case, *S* was found to 0.80, a relatively high value. The discrepancy between *S* determined by absorption and *S* found by emission probably arises either from the different measurement geometry or from the presence of the extra band in the perpendicular absorption. In the case of absorption, transmitted light is monitored whereas for fluorescence, it is light scattered from the sample surface which is detected.

Variation of Order with Temperature

In order to monitor changes in order as a function of heating, temperature dependent birefringence was measured using a hot stage and a polarizing microscope (equipped with a Leitz type *M* tilting compensator). This method was specifically used since thermal processes might affect the temperature dependent emission characteristics independent of order. Figure *5* shows the variation of

FIGURE *5* Temperature dependence **of** birefringence (circles - heating, **crosses** cooling).

birefringence with temperature. There is little or no change in order as temperature increases. The is due to the very rigid nature of the crosslinked liquid crystal network. Similar characteristics to this have been observed for other acrylate functionalised liquid crystal networks. [**111.**

It is also interesting to note that the birefringence returns to its original value on cooling to ambient temperature indicating that fluctuation of order is probably due to random thermal motion rather than relaxation of the order.

Raman Anisotropy Studies

In an attempt to gain greater insight into the degree and nature of ordering in these systems, we undertook Raman inisotropy studies.

Raman spectroscopy was chosen specifically because it was found that the carbon -carbon triple bond stretch from **I11** was too weak to be observed in the IR spectrum of cured films. The thin film form of the samples makes them much easier to measure using Raman spectroscopy than infrared. it was also found that the Raman spectra contains far fewer peaks and it is therefore simpler to interpret spectra and measure absolute peak intensities.

The studies were designed to probe the chemical nature of ordering in these systems. This was achieved by measuring the polarization dependence of Raman peaks which may be assigned discreetly to modes exhibited by either the C6H or the III moiety in the mixed, photopolymerised films.

In order to study order in the films, pump polarization dependent Raman spectra were recorded. To distinguish between modes associated with the host and fluorescent guest, the Raman spectra of the two monomers in powder form were first recorded (Figs. 6a and b).

The spectral region or interest here is from 1585 to 2250 cm^{-1} since here there are peaks which may be unambiguously assigned to either C6H or III molecular cores. For III, the peaks at 1590 and 2200 cm⁻¹ are of interest. These are assigned to a phenyl mode and a carboncarbon triple bond mode respectively. For C6H, the peaks at 1600 and 1730 cm^{-1} are assigned as phenyl ring and carbonyl modes respectively.

FIGURE *6* Raman spectra of the monomers **C6H** and **111**

FlGURE *6* (Continued)

It is interesting to note the absence of a peak at around 1730 cm^{-1} in the spectrum of **111** (which also contains carbonyl bonds in the acrylate functionalities). The presence of this peak in the spectrum of **C6H** is therefore probably from the ester linkages and not from the acrylate end groups.

Equivalent peaks to these at practically identical frequencies were found in cured film mixtures and thus it is assumed that the *in situ* photopolymerization process has no effect on the positioning of these peaks. Preliminary investigations showed that all four peaks shared a similar polarization dependent intensity variation.

The angular dependence of Raman scattering intensity was therefore investigated for these four peaks in a 10% III; 90% C6H in situ cured film. Raman spectra were recorded for various angles with respect to the orientation direction and the integrated intensity dependence for the four peaks is shown in Figures 7a and 7b. Figure 7a shows the angular dependence of intensity for the two peaks associated with **111** and Figure 7b shows those for **C6H.**

As expected, all of the peaks show a symmetrical, periodic variation of intensity with angle. This demonstrates ordering of both the guest and host in these films.

FIGURE 7a Angular dependence of intensity for peaks associated with **I11** (lines depict fits to Eq. (2)).

FIGURE **7b** Angular dependence of intensity for peaks associated with **C6H** (lines depict fits to Eq. (2)).

In order to compare the degree of order of **C6H** and **111** in the films, the order parameters *(S)* was determined from the measurements. *S* is defined here as:

$$
S = \frac{I_{\text{pa}} - I_{\text{pe}}}{I_{\text{pa}} + 2I_{\text{pe}}}
$$
 (3)

where I_{pa} and I_{pe} are respectively the peak intensities parallel and perpendicular to the orientation direction.

In order to determine values of I_{pa} and I_{pe} for the four peaks, the data shown in Figure 2 was fitted to the following function:

$$
I(\theta) = A\cos^2(\theta - B) + C \tag{4}
$$

where *A* is an amplitude term, *B* is a phase correction accounting for any discrepancy between 'internal' and 'external' angle and *C* accounts for finite intensity at minima. Equation **(3)** may now be rewritten as:

$$
S = \frac{A}{A + 3C} \tag{5}
$$

Values of *S* for four peaks were then determined from the fits (shown in Fig. 2) and are listed in Table I.

The values obtained are clearly consistent within errors. These results suggest a distinct difference in the degree of order of **C6H** and **I11** in the photopolymerized film. **C6H** shows a far higher degree of ordering than **111** perhaps due to the fact that the flexible side chains in **I11** are positioned either side of rigid core whereas in **C6H,** they are at either end of the long axis. This may limit the degree of ordering **Ill** can achieve in the mesophase and consequently in the photopolymerized film.

It has been shown previously [10] that the temperature of photopolymerization affects the order in **C6H** films. The polymerization

*Errors are derived from fits

temperature is usually expressed in terms of 'reduced' temperature (T_r) :

$$
T_{\rm r} = \frac{T_{\rm pol}}{T_{\rm cl}}\tag{6}
$$

where T_{pol} is the polymerization temperature and T_{cl} is the clearing temperature (or temperature at which transition to the isotropic phase occurs). To investigate the dependence of order on reduced temperature, Raman studies similar to those already outline were carried out on 5% **III** cured films with T_r values of 0.7, 0.8 and 0.9. For convenience, only two peaks were considered; 1730 cm⁻¹ (C6H) and 2210 cm^{-1} (III). The findings of these studies are summarized in Table 11.

As might be expected, there appears to be a downward trend in the order of both **C6H** and **III** as T_r increases (and the isotropic phase is approached). Again, it is clear that the **I11** is not as highly ordered as the **C6H**. It seems likely that prior to polymerization, **C6H** imparts order to **111** which is then 'frozen in' when *in situ* polymerization occurs.

The results of this study indicate possible reasons for the discrepancy in the previous absorption and photoluminescence measurements. Since **111** and **C6H** appear to be ordered to different degrees, it might be expected that there would be variable contributions from each to the absorption and photoluminescence anisotropy.

EXPERIMENTAL

Differential Scanning Calorimetry (DSC) Measurements

Measurements were performed on Perkin Elmer **DSC-4.** The sample quantities were in the range of 1Omg. The heating and cooling rates

S^*	$CH(1730 \, cm^{-1})$	$III(2210 cm^{-1})$
$T_r = 0.7$	0.85 ± 0.18	0.35 ± 0.09
$T_r = 0.8$	$0.84 + 0.18$	0.32 ± 0.03
$T_r = 0.9$	$0.67 + 0.11$	0.26 ± 0.08

TABLE 11 Order parameter values as a function of reduced temperature

* Errors **are** derived from tits.

were 10°C per minute, the measurements were carried out under an inert nitrogen atmosphere.

Thin Film Preparation

Oriented photopolymerized thin films were produced by an oriented rubbing method. A liquid crystal display type glass cell of $10 \mu m$ thickness was fabricated. The inside walls of the cell were coated with a thin layer (ca. $0.1 \mu m$) of spun cast Nylon 66 which was rubbed along one direction of the film plane in order to induce alignment when the glass cell was filled with liquid crystal material. Long range ordering could not be induced in samples of compound **111.** Thus ordered thin films of **I11** could not be produced.

Compound **I11** *(5%* by weight) was mixed with 1,4-phenylene **bis{4-[6-(acryloyloxy)hexyloxy]** benzoate) 2% mol. of photoinitiator $(CIBA-IrgacureTM 651)$ and 0.1 mole percent of thermal inhibitor (4-methoxyphenol) was added.

A glass cell containing the mixture was brought to 130°C (nematic phase) and photopolymerized by irradiation for 20 minutes with a low intensity UV fluorescent lamp (4 W) lamp. The cross-linked film thus produced is of very good optical quality and exhibits no sign of phase separation or photodegradation. The same film was used in all subsequent studies.

Absorption and Fluorescence Measurements

Polarized absorption spectra were obtained using an AT1 UV/visible absorption spectrometer and a Rowi 55 mm polariser. The polarizer was placed between the spectrometer source and the film sample. The parallel absorption spectrum was recorded with the direction of polarization parallel to the rubbing direction of the film. The polarizer was rotated 90° and the perpendicular absorption spectra was recorded. Polarized flourescence spectra were measured using a Perkin Elmer MPF-4413 spectrophotometer. The spectrophotometer source was intrinsically polarized so no polarizer was required. The parallel and perpendicular spectra were recorded by aligning the film at the appropriate angle (0° or 90°) with respect to the polarized source. The film was placed at an angle of 45° to both the excitation source and detector. The excitation wavelength was 350 nm.

Birefringence Measurements

Measurements were performed using a Leitz polarizing microscope and a Leitz tilting compensator (type M @ 546 nm). The samples were heated using a microscope hot stage and a Eurotherm temperature controller.

Raman Spectroscopy Measurements

Raman spectra were recorded using an Instruments **S.** A. LABRAM 1 **B** confocal microprobe system operating with a polarized Helium Neon laser at a wavelength of 632.18 nm. The polarization extinction ratio of the laser was measured to be greater than 500:l. A 50X objective was employed, giving a nominal spot size of $1.2 \mu m$. The power incident on the sample was IlmW. Polarization angle dependent spectra were recorded by rotating the sample in the plane perpendicular to the incident beam. The objective was focused before each new spectrum was recorded to ensure completely consistent incident power levels were achieved.

Preparation of the Fluorescent Compound

All reactions were performed under an Argon atmosphere. All solvents were dried and degassed before use. Reagents were used as supplied from the Aldrich chemical company. NMR spectra were recorded in CDCl₃ solution with an internal TMS standard.

Synthesis of $II - 1$ mmol of I and 2.1 mmol of phenylacetylene were dissolved in 30 ml of piperidine. 0.046 g (2 mol\%) of Pd(PPh₃)₄ and 4 mg of Copper (I) iodine were then added and the mixture was stirred at 90°C for 6 hours. Following cooling, the precipitated hydrobromide salts were filtered and washed with hexane. The washings were combined with the piperidine solution and the solvents were removed under vacuum. The resulting solid was recrystallized from butanone. Yield 0.42 g (83%).

 ${}^{1}H = NMR: \quad \delta = 0.92 - 1.80$ (m, 8H, $- (CH₂)₄$); 3.78 (t, 2H, $-OCH_2$, 3.83 (t, 2H, HO-CH₂-), 6.81 (s, 1H, Ar-H), 7.20 – 7.60 (m, 5H, Ar-H) ppm. ¹³C NMR δ = 22.5, 25.4, 29.8, 32.0 (CH₂); 69.9 ($-OCH_2$), 72.4 (HOCH₂), 86.3, 95.0 (spC), 114.2(Ar C), 117.1(Ar C), 123.4(Ar C), 128.1 (Ar C), 132.0(Ar C), 154.0 (Ar C) ppm.

Synthesis of III:-0.5 mmol of II was dissolved in 20 ml of THF along with 3mg of hydroquinone stabiliser and 1.1 mmol of triethylamine. The solution was heated to reflux and **I** .1 mmol of methacroyl chloride dissolved in 5 ml of $CH₂Cl₂$ was gradually added. Stirring was continued for 2 hours before the solution was allowed to cool to room temperature. The mixture was then poured into 150 ml of water and washed with saturated sodium carbonate solution $(3 \times 20 \text{ ml})$, dried and evaporated under vacuum. The solid was recrystallized from ethanol. Yield 0.23 g (60%).

¹H NMR: $\delta = 1.52 - 1.85$ (m, 8H), 4.00 (t, 2H), 4.18 (t, 2H), 5.02 $(a, 3H)$, 5.81 (d, 1H), 6.13 (d, 1H), 6.80 (s, 1H), 7.21 - 7.62 (m, 5H) ppm. ¹³C DEPT NMR: $\delta = 25.71$ (CH₂), 28.53 (CH₂), 29.07 (CH₂), 114.21 (Arc), 117.24(ArCH), 124.16(ArCH), 128.32(ArCH), 131.24 $(ArCH)$, 146.59 (CH), 147.20 (CH₂), 153.98 (ArC--O), 159.92 (C==O) ppm. 64.48 (CH₂), 67.95 (CH₂), 86.20 (C \equiv C), 94.87 (C \equiv C), 101.20 (CH₃),

CONCLUSION

Thin films of a new oriented, cross-linked liquid crystal mixture have been shown to exhibit strongly polarised absorption and emission and large order parameter values. Furthermore, temperature dependent birefringence measurements indicate that order in the sample is largely maintained at elevated temperatures.

It was also noted that the birefringence (and hence order) returned to its original value on complete cooling. Measurements of the order parameter from absorption and emission provides large values. The discrepancy between these values is probably due to different measurements techniques or *to* the extra absorption feature in the perpendicular polarized absorption spectrum.

Order in these films was also probed using Raman anisotropy measurements. These studies show that the two different molecular components in the films are ordered to different degrees. The order parameter for each component has been determined for two Raman peaks and the results were found to be self-consistent.

The affect of photopolymerization temperature on order was also probed and it was found that as temperature increases, the degree of order of both molecular components decreases. The use of Raman spectroscopy has proven to be advantageous in this case where thin film samples are studied. There is also an advantage here due to the greater Raman activity of the carbon-carbon triple bond mode as compare to infrared spectroscopy.

Further studies on fabrication and testing of electrically driven devices are in progress with a view to producing a highly polarized light emitting diodes.

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