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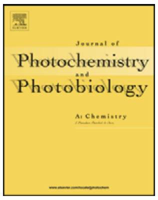

Intrinsic luminescence properties of ionic liquid crystals based on PAMAM and PPI dendrimers

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ABSTRACT

The photophysical properties of ionic liquid crystals based on poly(amidoamine) {PAMAM} and poly (propyleneimine) (PPI) dendrimers were examined both in solutions and in thin solid films. The dendrimers were peripherally modified by decanoic, 4-octyloxybenzoic, and 3,4,5-trioctyloxybenzoic acids to generate dendrimer based ionic liquid crystals of smectic and columnar phases. The liquid crystalline properties of these compounds were examined by polarizing optical microscopy (POM) and differential scanning calorimetry (DSC). The dendrimer based ionic liquid crystals exhibited blue emission upon excitation at 370 nm. The emission intensity was found to be enhanced by an order of magnitude in solution when ionic liquid crystals are made from higher generation dendrimers. The steady state and time resolved fluorescence experiments suggest that optical properties of the liquid crystals are originated from the intrinsic emission properties of amine terminated PAMAM and PPI dendrimers. The findings presented here show for the first time that PAMAM and PPI based mesogenic structures can exhibit intrinsic emission in the visible region without incorporating conventional fluorophores.

1. Introduction

The field of dendrimers constitutes one of the most important areas of frontier research [1–5]. While dendritic structures can be considered as a special class of polymers, lucidly defined structural features, sub-nanometer inner cavities and flexibility in incorporating functional groups to the periphery or interior parts provide distinct physical and chemical properties to these macromolecules compared to the classical polymers [6]. Numerous structural scaffolds for dendrimers are reported over the past two decades, ranging from pure organic molecular framework to organometallic as well as biomaterials [7]. Since synthetic strategies for various types of dendrimers have been optimized, recent efforts are more focused towards utilizing dendrimers for attractive material applications [8–10]. Dendrimer based mesogenic materials (liquid crystals) are of paramount importance in this context [11–15]. Introducing mesogenic properties to dendrimers largely depends on the topology of the branching units of the dendrimer. Two widely accepted methodologies to prepare dendrimer based liquid crystals are: (a) functionalize the periphery of dendrimers and (b) functionalize at the interior portions of the dendrimer by mesogenic units. One of the main attractive aspects of dendrimer based liquid crystals is the assimilation of characteristic properties of dendrimers into a mesophase, which is expected to provide alternate means to surmount the present challenges in the filed, especially such as low brightness and energy efficiency of liquid crystal based display systems.

Recent developments in dendrimer field suggest that certain amine based dendrimers-poly(amidoamine) {PAMAM} and poly propyleneimine (PPI) dendrimers-exhibit an unusual luminescence in the visible region, in the absence of conventional fluorophores. Intrinsically fluorescing dendrimers was initially reported for carboxylate terminated PAMAM dendrimers [16]. Corroborating this work, Wang and Imae, reported an intense, blue emission from amine terminated, structurally un-modified PAMAM dendrimers at highly acidic conditions [17]. In a separate attempt, Bard and co-workers have reported the blue luminescence from chemically oxidized hydroxy terminated PAMAM dendrimers [18]. This work was followed by numerous other reports describing the interesting blue emission from dendritic as well as linear polymeric chains in the absence of conventional fluorophores [19–21]. The following aspects were found to be crucial in controlling the intrinsic emission intensity from PAMAM and PPI dendrimers: (a) pH of the medium; (b) extensive hydrogen bonding;(c) air oxidation leading to luminescent moieties; (c) aggregation or self-assembly of dendrimers in solution phase; and (d) electron–hole recombination. While the underlying mechanistic reasons of intrinsic emission from PAMAM, PPI and similar types of dendrimers/polymers have not been unraveled completely, the bright, blue emission has remained as an attractive property for many practical applications. We hypothesized that blue emitting luminescent liquid crystals

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can be generated by peripheral modification of intrinsically fluorescing PAMAM or PPI dendrimers, provided the intrinsic emission property can be retained in the mesophase.

In the present study, we examined the steady state luminescence intensity, excited state lifetimes, fluorescence quantum yields and thin film fluorescence of a series of PAMAM and PPI based ionic liquid crystals (Chart 1). The peripheral units utilized to generate the mesogenic properties in the dendrimers are decanoic, 4-octyloxybenzoic, and 3,4,5-trioctyloxybenzoic acids which have been linked to the periphery of PAMAM and PPI dendrimers through ionic interactions. The intrinsic emission properties of PAMAM and PPI dendrimers are found to be present in the liquid crystal phase as well, providing a straight forward method to generate luminescent liquid crystals.

2. Experimental

2.1. Materials

The higher generation dendrimers were purchased from Aldrich Chemical Co. as 10 wt% solution in methanol and the solvent was evaporated under inert atmosphere prior to dissolving in other solvents. Experiments were done with distilled deionized water (Millipore filtration system). Decanoic acid was purchased from Merck. 4-Octyloxybenzoic acid and 3,4,5-trioctyloxybenzoic acid were synthesized using reported procedures. Lower generation dendrimers were also synthesized using standard procedures.

2.2. Methods of preparation

As a representative case, preparatory method of compound **III** from Chart 1 is described below. First generation amine terminated PPI dendrimer (0.10 g, 0.30 mmol) was added to a solution of 3,4,5-trioctyloxybenzoic acid (0.70 g, 1.38 mmol) in dry THF. The mixture was sonicated for 10 min in a bench top sonicator and then the solvent was evaporated in a rotary evaporator under vacuum and further dried by purging a stream of inert argon. The nearly complete conversion of the starting materials to the ammonium-carboxylate salt was confirmed by IR spectroscopy. The complete disappearance of the acid carboxyl peak at 1710 cm^{-1} and the appearance of the asymmetric carboxylate peak at 1557 cm^{-1} were observed confirming the formation of the ionic salt. Similar procedure was adopted for the preparations of compounds **I**–**VII**.

2.3. Instruments

UV–vis experiments were performed on a PerkinElmer Lambda 25 UV–vis spectrophotometer. Luminescence experiments were carried out using a Hitachi F4500 fluorescence spectrophotometer. The fluorescence decay measurements were carried out using the time correlated single photon counting technique (TCSPC) with micro channel plate photomultiplier tube (MCP-PMT) as detector and picosecond laser as the excitation source (model 5000 U, IBH, UK). The optical textures of the mesophase were studied with a Nikon Eclipse LV100 POL polarizing microscope with manually made heating and cooling chamber. The transition temperatures and enthalpies were determined by differential scanning calorimetry (DSC) with a NETZSCH DSC 204 instrument operated at a scanning rate of 10 °C\min under nitrogen atmosphere.

3. Results and discussion

3.1. Mesogenic properties of dendrimer based ionic liquid crystals

There has been a sustained interest to generate luminescent liquid crystals due to its prevalent applications in various fields such as emissive display systems [22]. One of the hurdles in the design and synthesis of luminescent liquid crystals is the loss of emission properties of the incorporated fluorescent molecules due to their close proximity in a close range order in the mesophase. This has been referred to as 'self-quenching' of the excited fluorophores due to immediate interactions between them in the mesophase. It was recently found, however, that careful design leading to specific molecular arrangements can generate enhanced fluorescence from certain pyrene based discotic liquid crystals and J-type aggregates in columnar ordering [23,24]. Another elegant study by Park and co-workers have shown that excited state intramolecular proton transfer (ESIPT) can be utilized to develop fluorescent liquid crystals with large Stoke's shift (173 nm) in fluorescence emission [25]. They have also demonstrated that a few oxidiazole based ESIPT molecule, which emits orange fluorescence, can be used as an emitting layer in white light emitting OLED devices upon combining with complementary component.

The synthesis and mesogenic properties of PAMAM and PPI derived ionic liquid crystals have extensively been studied by Serrano and co-workers [26,27]. Nonetheless, the photophysical properties of these materials have not been investigated, albeit the parent dendrimers posses interesting luminescence properties. We have initiated examining the intrinsic emission properties of liquid crystals which are generated through peripheral modification of PAMAM and PPI dendrimers. The structure of PAMAM and PPI dendrimer based ionic liquid crystals utilized in this study are shown in Chart 1.

The liquid crystalline properties of **I**–**VI** were examined by polarizing optical microscopy (POM) and differential scanning

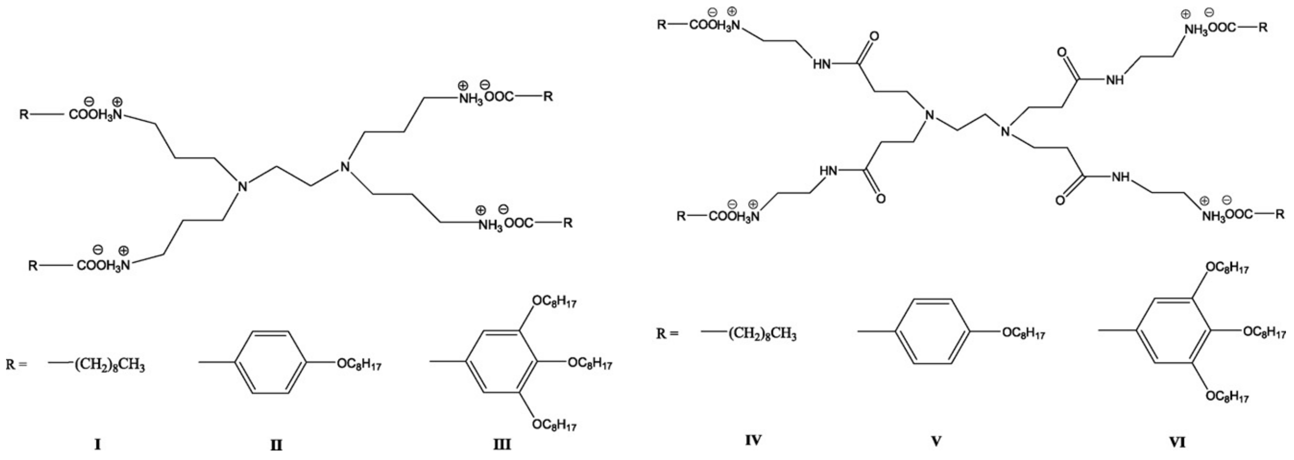

Chart 1. Structures of PAMAM and PPI based mesogenic compounds utilized in the study.

calorimetry. Compound **I** shows Smetic A phase as reported previously [26]. While compound **II** exhibited Smectic A phase similar to its higher homologues derivative (PPI peripherally modified by decyloxybeznoic acid), the phase transition was relatively at a lower temperature. This is consistent with the previous observations that decrease in length of alkanoate moiety results in the decreased phase transition temperature. Compound **III** exhibited columnar texture. All the three PAMAM based ionic liquid crystals (**IV**–**VI**) showed Smectic phase above room temperature. Differential scanning calorimetric studies were carried out for the dendrimer based liquid crystals in order to confirm the phase transitions observed in the POM studies.

3.2. Aggregation properties of dendrimer based ionic liquid crystals

The luminescence properties of compounds **I**–**VI** were initially examined in solution. It has been reported in the literature that peripherally modified, higher generations of PAMAM dendrimers form unimolecular micelles-which are concentration independent-in non-polar solvents such as toluene [28]. On contrary, lower generation PAMAM dendrimers form concentration dependant micelles which largely depend on the nature of the peripheral groups. Studies by Jia and co-workers suggested that lower generation PAMAM dendrimers, peripherally modified by pyrene, naphthalene, and perylene, tend to aggregate in solution attaining 'twin' type micellar structures, which exemplifies concentration dependant micelle formation [29]. Critical aggregation concentrations (CAC) for such dendrimers were found to be in between 10^{-7} and 10^{-5} M. Interestingly, Meijer and co-workers have found that lower generation PAMAM dendrimers peripherally modified by long alkyl chains do not aggregate in toluene [30].

In order to verify whether the compounds used in the present study exhibit aggregation properties in solution phase, we have prepared the solutions of **I**–**VI** in toluene and found that **II**, **IV** and **V** have limited solubility in toluene. This corroborated the findings of Meijer, indicating that the micelle formation, which leads to the solubility of these ionic compounds in non-polar solvent like toluene, is restricted in the lower generation of PAMAM or PPI dendrimers. Nonetheless, compounds **I**, **III** and **VI** were fairly soluble in toluene. This could be presumably due to the increased number of non-polar chains per molecule, especially in **III** and **VI** which might result in the enhanced hydrophobic interactions.

3.3. Photophysical properties of dendrimer based ionic liquids

Interestingly, the steady state emission spectra of these compounds resemble to that of the intrinsic emission spectra of corresponding dendrimers. The intrinsic emission of PAMAM and PPI dendrimers is reported to have a $\lambda_{\max}$ of 470 nm [17]. All the ionic liquid crystals examined in the present study exhibited emission in the blue region of the spectrum and the features of the excitation and emission spectra were identical to that of the reported ones for the dendrimers. This suggests that the intrinsic emission from the dendrimers were not hampered by the peripheral modifications. The excitation and emission spectra of **III** are shown in Fig. 1.

In order to verify whether concentrations of lower generation dendrimers are dependant on the intrinsic emission intensity, dependence of a wide range of concentration of **III** on its intrinsic emission intensity was monitored in toluene. Fig. 2 depicts the variation of emission intensity upon changing the concentration of **III**, which is clearly linear in the initial part, reinforcing the above fact that lower generation dendrimers do not aggregate in the solution state.

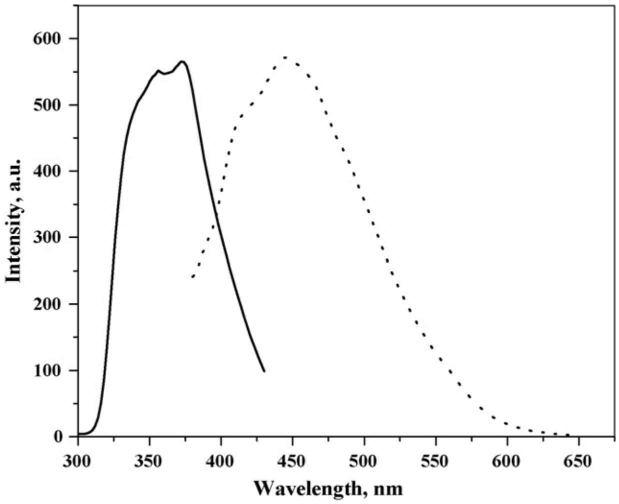

Fig. 1. Excitation and emission spectra of **III** (3.2 mM) in toluene. The emission maximum was 444 nm.

While some of the compounds were not soluble in toluene, all of them were soluble in methanol. The solubility of these compounds in polar solvents could be due to the presence of polar groups in PAMAM dendrimer as well as the ionic groups present in the systems. The excitation and emission spectra of **III** in methanol are given in Fig. 3. Inset of Fig. 3 contains a plot which compares the steady state luminescence intensity of all the compounds used in the present study in methanol. The plot suggests that **VI** emits with highest intensity. The luminescence quantum yield of **I**–**VI** was determined in toluene and methanol using coumarin as a reference and the values obtained are given in Table 1 [31]. The quantum yield values were calculated utilizing the following equation:

$$\Phi_{\mathrm{S}} = \frac{\Phi_{\mathrm{R}} f_{\mathrm{S}} A_{\mathrm{R}} n_{\mathrm{S}}^{2}}{f_{\mathrm{R}} A_{\mathrm{S}} n_{\mathrm{R}}^{2}},$$

where, Φ is the quantum yield, f is the area under the emission spectrum, A is the optical density, n is the refractive index of the medium, S and R represents the sample and the reference respectively. The quantum yield values also suggest that **VI** emits in relatively high intensity. This is consistent with the previous observations by Imae and co-workers that PAMAM emits higher in intensity than the corresponding PPI dendrimers [19]. This could be due to the fact that

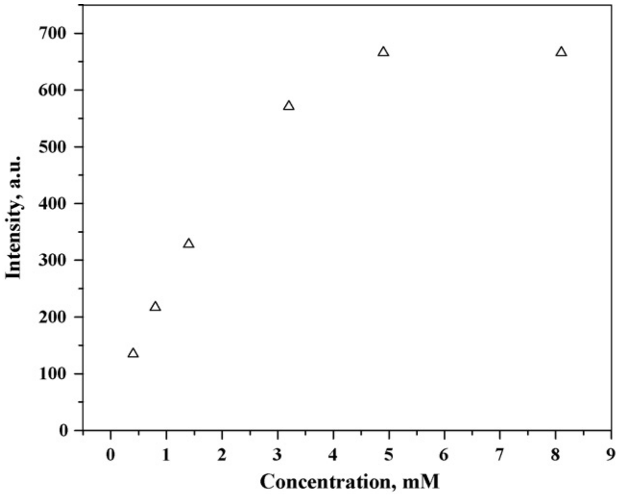

Fig. 2. Dependence of concentration of **III** on the intrinsic emission intensity in toluene.

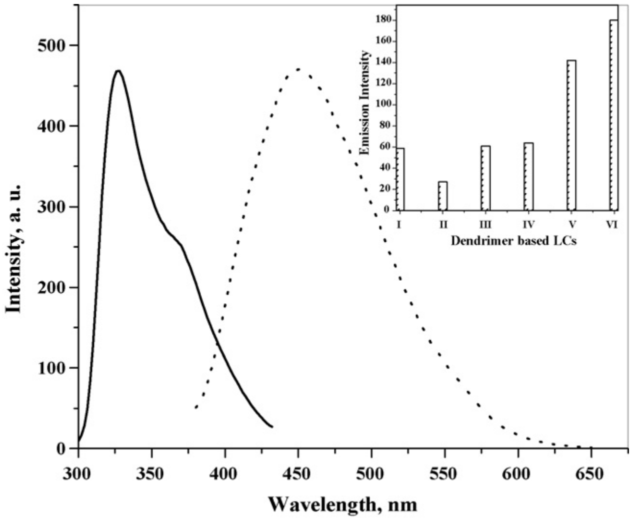

Fig. 3. Excitation and emission spectra **III** (1.4 mM) in methanol. Inset shows the emission intensity of compounds **I**–**VI**, excited at 370 nm with identical optical density.

Table 1
Photophysical properties of PAMAM and PPI dendrimer based ionic crystals.

System	Emission* λ_{max} (nm)	Excited state lifetimes in toluene and methanol (ns)*	Quantum yield[a,*]
I	448 (455)	2.6 (3.0), 7.0 (8.2)	0.02 (0.02)
II	(–) (435)	(–) (3.2), (–) (9.3)	(–) (0.01)
III	441 (450)	2.8 (2.5), 8.3 (7.7)	0.04 (0.02)
IV	(–) (448)	(–) (3.4), (–) (8.0)	(–) (0.02)
V	(–) (454)	(–) (4.2), (–) (7.9)	(–) (0.04)
VI	450 (456)	2.6 (3.0), 7.4 (6.5)	0.05 (0.04)
VII	453 (–)	2.7 (–), 7.5 (–)	0.10 (–)

(–) The values are not measured due to limited solubility.

* Values of methanol are given in parenthesis.

[a] Error bar of 5%.

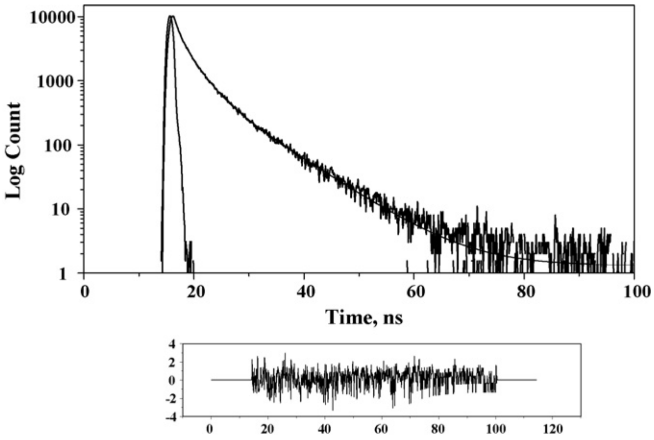

Fig. 4. Excited state decay of **III** in methanol. Excitation wavelength was 370 nm.

PAMAM derivatives have enhanced rigidity, compared to the PPI based compounds, due to the hydrogen bonding present between the carbonyl and amine functionalities.

The excited state lifetime of **I**–**VI** were examined in toluene and methanol and the values are given in Table 1. Fig. 4 contains the excited state decay of **III** in methanol. Previous reports have shown that PAMAM and PPI dendrimers exhibited double exponential excited state decays, irrespective of the terminal groups [16,17]. Compounds **I**–**VI** also exhibited double exponential decays, consistent with the hypothesis that the emission properties of the dendrimer based ionic liquids originated from their parent dendrimers. The lifetime value of the short component was close to 3 ns and that of the long component was around 8 ns. The origin of the excited state decays in PAMAM or PPI dendrimers is still not assigned explicitly as the mechanism of the intrinsic emission is not well established. While the intensity of the steady state fluorescence from PAMAM and PPI dendrimers varies upon altering the solvent medium as well as peripheral groups, the excited state lifetime values remained identical within the experimental error for

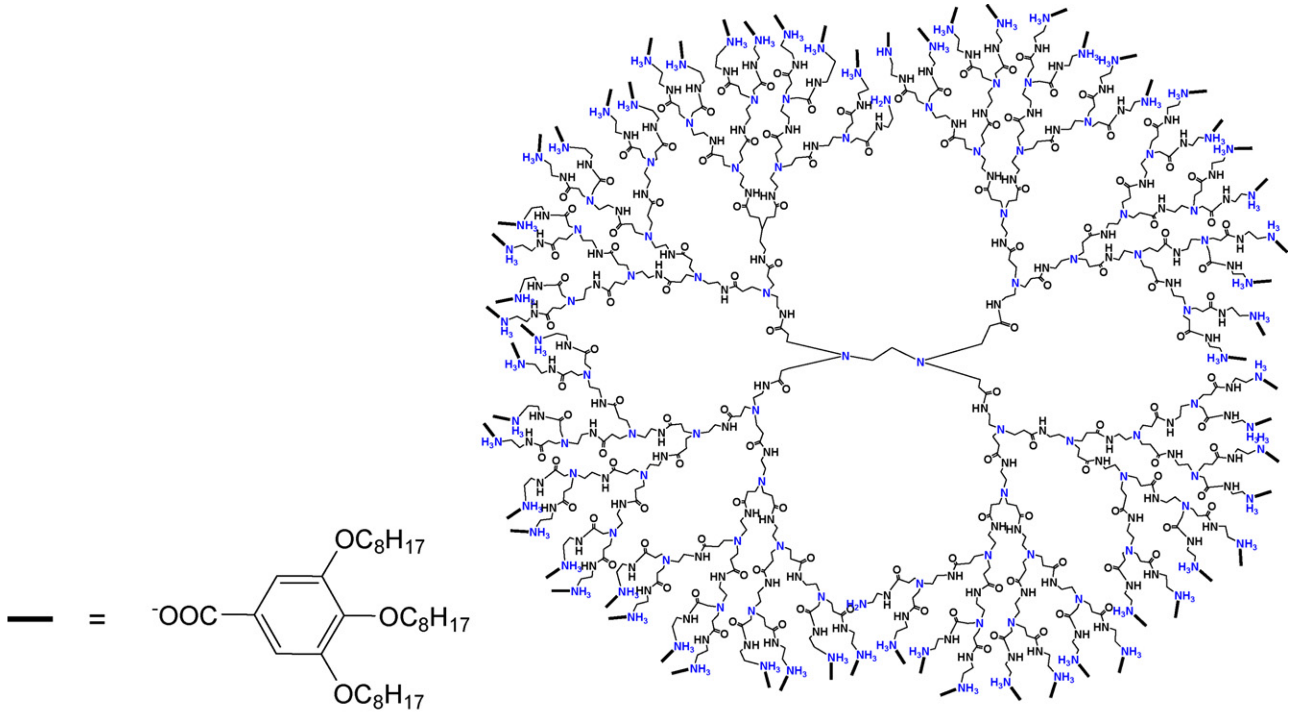

Chart 2. Structure of **VII**.

all the systems examined in both methanol and toluene. The obvious inference is that enhanced emission observed upon changing the solvent is due to the decreased non-radiative pathways of the intrinsic emissive state present in the dendrimeric systems.

It has been previously reported that the intrinsic emission from higher generation PAMAM dendrimers is relatively intense compared to its lower generations [17]. For example, Imae has shown that a fourth generation PAMAM dendrimer emits with almost four times more intense compared to that of a second generation PAMAM dendrimer at identical conditions. This suggests that luminescent liquid crystals with enhanced emission intensity can be generated by peripherally modifying higher generation dendrimers having enhanced intrinsic emission intensity. To verify this effect in the PAMAM derived ionic liquid crystals, fourth generation PAMAM dendrimer (64 number of peripheral units are present in a fourth generation PAMAM dendrimer) was peripherally modified by 3,4,5-trioctyloxybenzoic acid and the luminescence properties were examined (**VII**) (Chart 2). This particular derivative was chosen because of the enhanced intrinsic emission intensity introduced by 3,4,5-trioctyloxybenzoic acid moiety in **III** and **VI**. As the peripherally modified fourth generation PAMAM dendrimer was insoluble in methanol, quantum yield experiment was performed in toluene. The quantum yield value obtained for **VII** in toluene was 0.10 ± 0.01, with coumarin as the standard. This is an order of magnitude higher than the corresponding value obtained for some of the lower generation dendrimers.

3.4. Thin film fluorescence studies of dendrimer based ionic liquid crystals

Next, the intrinsic fluorescence emission from PAMAM and PPI dendrimer based ionic liquid crystals is examined in thin solid film. The liquid crystalline material was taken in between two glass plates, which are thoroughly cleaned prior to the experiment. Except for **I** and **III**, which are room temperature liquid crystals, all other compounds were heated to their respective phase transition temperatures and annealed to room temperature to retain the mesophase in the thin solid film. Fig. 5 shows the emission spectra of compound **III** in the thin film.

Thin film luminescence spectra of compounds **I**–**VII** exhibited typical broad emission pattern, of which the emission spectrum of **VI** was wide enough to cover the entire wavelength from 380 to 600 nm (please see the supporting information). This suggests that **VI** can be incorporated into materials which can be used as white light emitters. The intense emission peak from the compounds also suggests that additional fluorophores are not needed in the system to obtain emission in the visible region. It is worth mentioning here that these ionic liquid crystals retained their emission properties intact in the thin solid film for a long period of time, demonstrating their stable material nature.

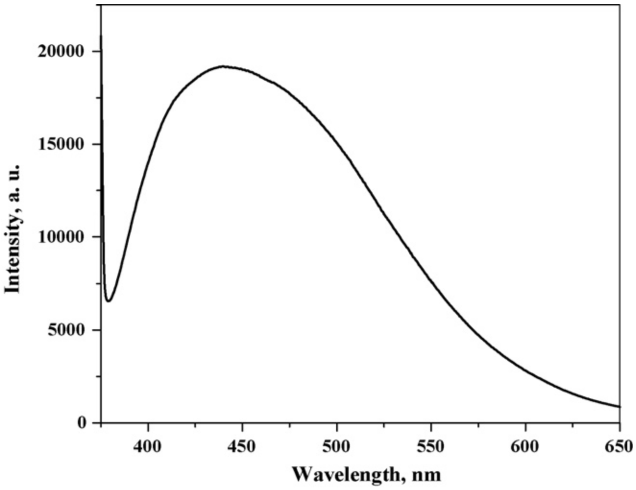

Fig. 5. Thin solid film spectrum of **III**. Excitation wavelength was 370 nm.

4. Conclusion

The intrinsic emission properties of PAMAM and PPI based ionic liquid crystals have been examined in solution and in thin solid film. The experimental results suggest that intrinsic emission properties of PAMAM and PPI dendrimers can be retained in the mesophase to generate luminescence liquid crystals in the visible region, without introducing conventional fluorophores. While incorporating rigid fluorophores in the mesophase quite often alters the phase transition temperatures of liquid crystal materials to higher values, utilizing the intrinsic emission properties of amine terminated PAMAM and PPI dendrimers in the mesophase provides an attractive alternative to prepare luminescent liquid crystals close to room temperatures. Attempts to enhance the emission quantum yield from PAMAM and PPI dendrimer based ionic liquid crystals are currently being initiated and the results of these studies will be reported in due course.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jphotochem.2008.12.023

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