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14. ABSTRACT In this grant period we have continued our studies of the synthesis, characterization and device fabrication of electro-optically active chromophoric macromolecules. The systems studied focused on novel conjugated main chain structures with high photoluminescent and electroluminescent quantum yields. The structures incorporated fluorene containing moieties, ethynylene bonds, terphenylene derivatives, <i>meta</i> and <i>ortho</i> variants of the paraphenylene unit, and a number of derivatized phenyl units either alone or in combination. In most cases the photophysical properties of the new materials were extensively studied. A new method to prepare efficient anode surfaces for devices using indium tin oxide nanoparticles was developed. Over 25 publications dealing with electrooptical polymers were published in the grant period. We have also continued our study of the coil-globule transition in solvated polar macromolecules focusing on temperature regimes just below the Θ -point. Other studies completed in the grant period included those involving surface modifications of polymer films by ion implantation orientated towards enhanced permselectivity, and a continuation of research studying ternary polymer blends in which one or more constituents is a copolymer. Altogether, some 36 manuscripts were published in the grant period, with a further 4 in press.			
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FINAL TECHNICAL REPORT

ELECTRO OPTICAL PROPERTIES
OF COPOLYMER BLENDS: LASING, ELECTROLUMINESCENCE AND
PHOTOPHYSICS

GRANT AFOSR F49620-03-01-0059

Period Covered: 1/15/03 – 12/31/05

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VI. ASTRACT OF ACCOMPLISHMENTS

In this grant period we have continued our studies of the synthesis, characterization and device fabrication of electro-optically active chromophoric macromolecules. The systems studied focused on novel conjugated main chain structures with high photoluminescent and electroluminescent quantum yields. The structures incorporated fluorene containing moieties, ethynylene bonds, terphenylene derivatives, *meta* and *ortho* variants of the paraphenylene unit, and a number of derivatized phenyl units either alone or in combination. In most cases the photophysical properties of the new materials were extensively studied. A new method to prepare efficient anode surfaces for devices using indium tin oxide nanoparticles was developed. Over 25 publications dealing with electrooptical polymers were published in the grant period. We have also continued our study of the coil-globule transition in solvated polar macromolecules focusing on temperature regimes just below the Θ -point. Other studies completed in the grant period included those involving surface modifications of polymer films by ion implantation orientated towards enhanced permselectivity, and a continuation of research studying ternary polymer blends in which one or more constituents is a copolymer. Altogether, some 36 manuscripts were published in the grant period, with a further 4 in press.

VII. DESCRIPTION OF RESEARCH

The grant has supported several research themes in the physical chemistry of macromolecules, including some that have continued from earlier AFOSR funding. The results have in large part been published (40 papers published and in press) in the open literature, and representative highlights are discussed below.

A. Electro-optically Active Polymers (1-3, 5, 7-9, 11-18, 21,22, 24, 26-34, 36-40)¹

We have pursued an integrated research program in this area in which new polymers have been a) synthesized b) characterized, especially with respect to their photo-physical properties and c) fabricated into devices such as light emitting diodes. Examples are given below.

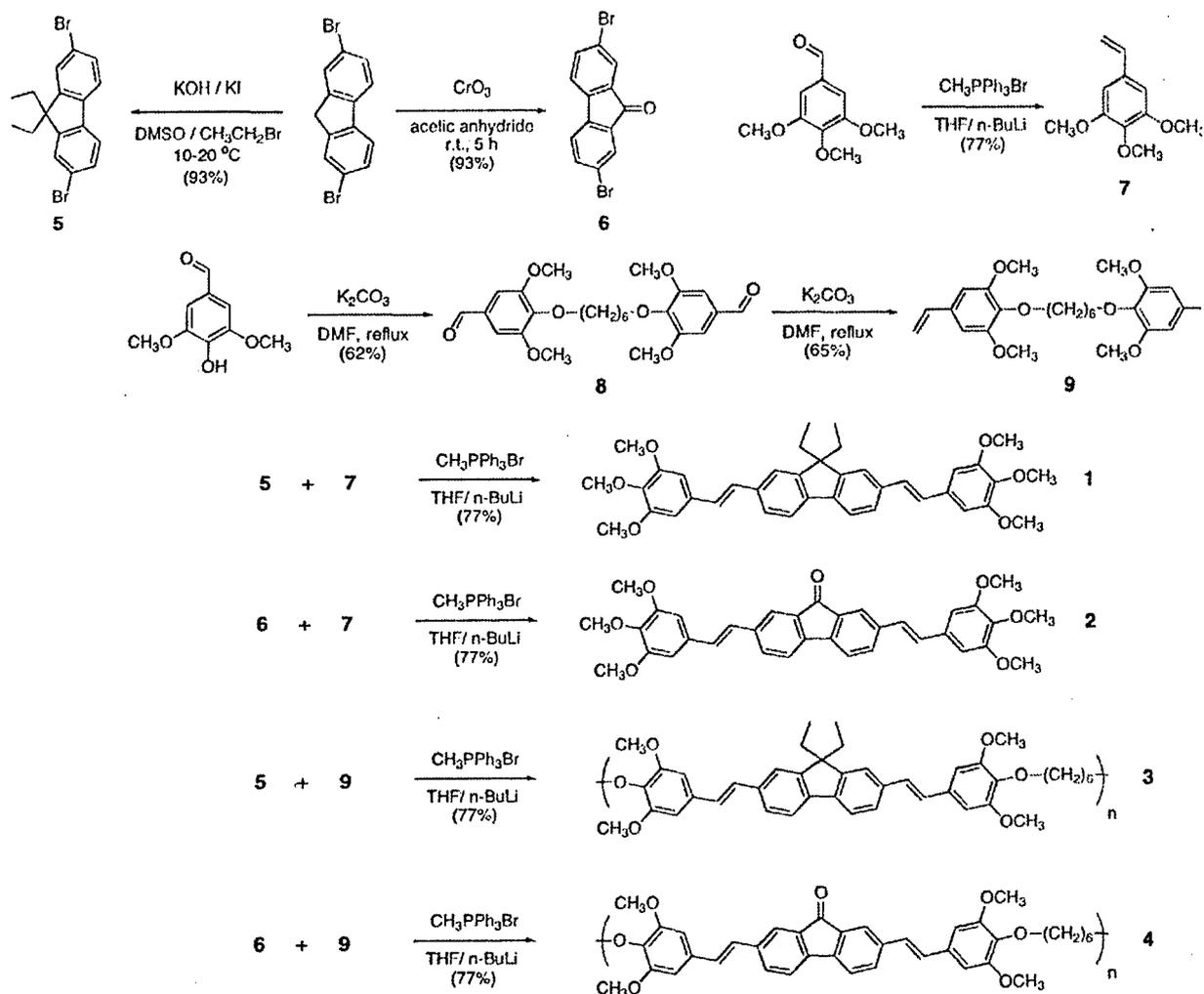
This group has carried out numerous studies of electroluminescent (EL) properties of segmented copolymers comprised of well-defined emitting PPV-type chromophores alternating with polymethylene chains. Such systems combine chromophore emission monodispersity with processibility due to the flexible polymethylene chain. One example is systems incorporating the quantum efficient fluorine group. The properties of segmented copolymers that incorporate fluorenes were compared to the homo-PPV type systems previously studied. An objective was to control characteristics in these segmented copolymers and corresponding model chromophores and then to compare these with fully conjugated fluorene and fluorenone-incorporating systems. In particular it was hoped to minimize or avoid formation of the undesirable long-wavelength emission bands formed in many dialkylfluorene-based emitters.

In this research (E,E)-2,7-bis(3,4,5-trimethoxyphenylethenyl)-9,9-diethylfluorene, (E,E)-2,7-bis(3,4,5-trimethoxyphenylethenyl)fluorenone, **1,2**, and their corresponding alternating segmented copolymer analogues *alt*-poly(2,6-dimethoxyphenylene-4-vinylene-[9,9-diethylfluorene-2-yl-7-vinylene]-3,5-dimethoxyphenylene-4-[1,6-hexanedioxy]) and *alt*-poly(2,6-dimethoxyphenylene-4-vinylene-[9-fluorenone-2-yl-7-vinylene]-3,5-

¹ Numbers refer to publications listed in Section VIII, below.

dimethoxyphenylene-4-[1,6-hexanedioyl]), **3,4**, were synthesized by Heck coupling. Compound **1** and polymer **3** have photoluminescence (PL) emission maxima at 420-465 nm, with solution quantum efficiencies of 0.93 and 0.77 relative to 9,10-diphenylanthracene. Compound **2** and polymer **4** have PL maxima at about 440 nm, as well as excimer emission bands at about 570 nm that increase in intensity as their solution concentrations increase. Electroluminescent maxima (device configuration ITO/PEDOT:PSS/Polymer/Ca-Al) for **1-4** were 450 nm, 610, 480 (broad) and 615 nm, with luminance of 0.515, 0.019, 0.020 and 0.059 cd/A, respectively. Molecule **1** had the highest luminance with a maximum brightness of 450 cd/m² at 7 V. The fluorenone derivatives both had dominant excimeric EL, while the diethylfluorene-based systems have efficient monomeric chromophore EL with very little long-wavelength emission. The lack of an undesirable long wavelength emission in **1** and **3** is attributed to poor π -stacking of chromophores, based on a crystallographic study of **1**. By comparison, π -stacking is favorable in **2** and **4**, since excimer emission is present in test LEDs, even when the chromophores are diluted with PMMA.

Scheme 1. Synthesis of **1-4**.



Model chromophore **1** and segmented copolymer analog **3** both exhibit blue solution phase photoluminescence with high quantum yields. They were utilized as emissive layers in organic LEDs and gave blue to blue-green emission, without formation of a long-wavelength emission component that is found in many dialkylfluorene-incorporating LEDs. This is attributed to a low tendency for π -stack formation between the fluorene units of chromophore **1** during film casting, thereby limiting or eliminating longer wavelength solid-state excimer emission. Fluorenone-containing model system **2** and corresponding

segmented copolymer **4** both show significant excimer emission in solution, and exclusive excimeric emission in the orange to orange-red region as emissive layers in test LEDs, even when diluted in a PMMA matrix as already noted. Presumably, π -stacking must be sufficiently favorable in the fluorenone chromophores to favor excimer generation as the dominant emission mechanism whenever sufficient molecules are present for aggregation. Blends of the fluorene-containing systems with the fluorenone-containing systems also gave predominant long wavelength emission consistent with fluorenone excimers, showing that the fluorenone chromophores aggregate in the film casting process even when mixed with fluorene-containing chromophores.

The results (Tables 1 and 2; Figs. 1, 4) show that, while long wavelength EL emission is observed in many fully conjugated polyfluorenes, it can be avoided in segmented copolymers that incorporate phenylene-vinylene-fluorene-vinylene-phenylene type chromophores. Fluorene π -stacking, which is a probable cause of this problem, can be avoided by judicious alteration of the fluorene-based chromophore to disfavor the π -stacking. By comparison, analogous chromophores that incorporate fluorenones allow excimer formation at higher concentrations even in solution, and nearly exclusive excimer emission in LEDs, even when the emissive layers are diluted by PMMA. These observations are significant in achieving better structure-property control of organic LEDs that incorporate fluorene and fluorenone-based chromophores.

Table 1. Selected photophysical properties of 1-4.

Sample	Solution ^a UV-vis λ_{\max} (nm)	Solution ^a PL λ_{\max} (nm)	Film ^b PL λ_{\max} (nm)	Solution Φ_{PL}^c	EL ^d λ_{\max} (nm)	EL CIE Coordinates ^{d,e}
1	384	440, 465	458	0.93	450	0.158, 0.384
2	314, 375, 475(br,wk)	442*, (565)*	430(wk), (600)	n/a ^f	610	0.501, 0.336
3	357	420, 440	469	0.77	480	0.182, 0.405
4	311, 355, 460(br,wk)	443*, (570)*	430(wk), (600)	n/a ^f	615	0.493, 0.334

^aChloroform solution; sh=shoulder, wk=weak, br=broad, *=concentration dependent, ()=excimer peak. ^bNeat film on quartz.

^cSolution results relative to 9,10-diphenylanthracene standard (ref 6). ^dITO/PEDOT:PSS/(emissive layer)/Ca-Al configuration.

^eEstimated using the 1964 scale of reference 13. ^fNot evaluated, due to variable behavior with concentration

Table 2. Selected LED properties using 2 in PMMA with comparison to LEDs using pure 1-4 as emissive layers.

Emissive Layer Composition (w/w %)	EL ^a λ_{\max} (nm)	Turn-on voltage (V)	Maximum luminance (cd/A)
1 (100%)	450	3	0.515
2 (100%)	610	4	0.019
2 (75%) ^b	600	4.5	0.045
2 (50%) ^b	591	5	0.190
2 (25%) ^b	589	6	0.160
2 (10%) ^b	570	12	0.033
3 (100%)	485	5	0.020
4 (100%)	610	4	0.059

^aITO/PEDOT:PSS/(emissive layer)/Ca-Al configuration. ^bw/w% diluted in PMMA (MW = 15,000).

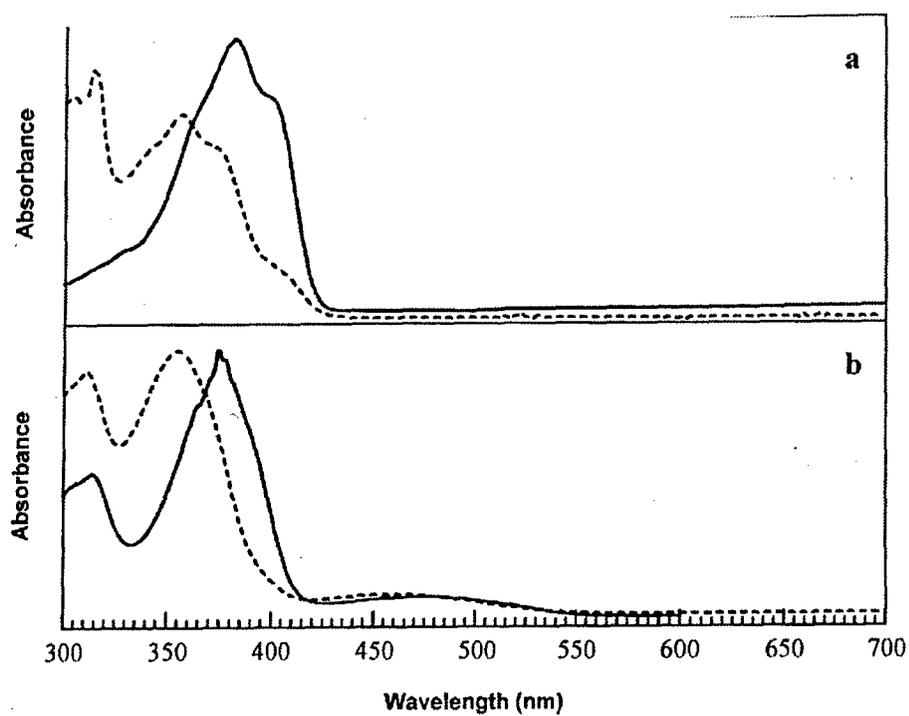


Figure 1: Comparison of UV-vis spectra for **1** (—) and **3** (- - -) in chart (a); for **2** (—) and **4** (- - -) in chart (b). All spectra in chloroform at room temperature, scaled on the ordinate relative to one another in each chart for ease of comparison of peak maxima.

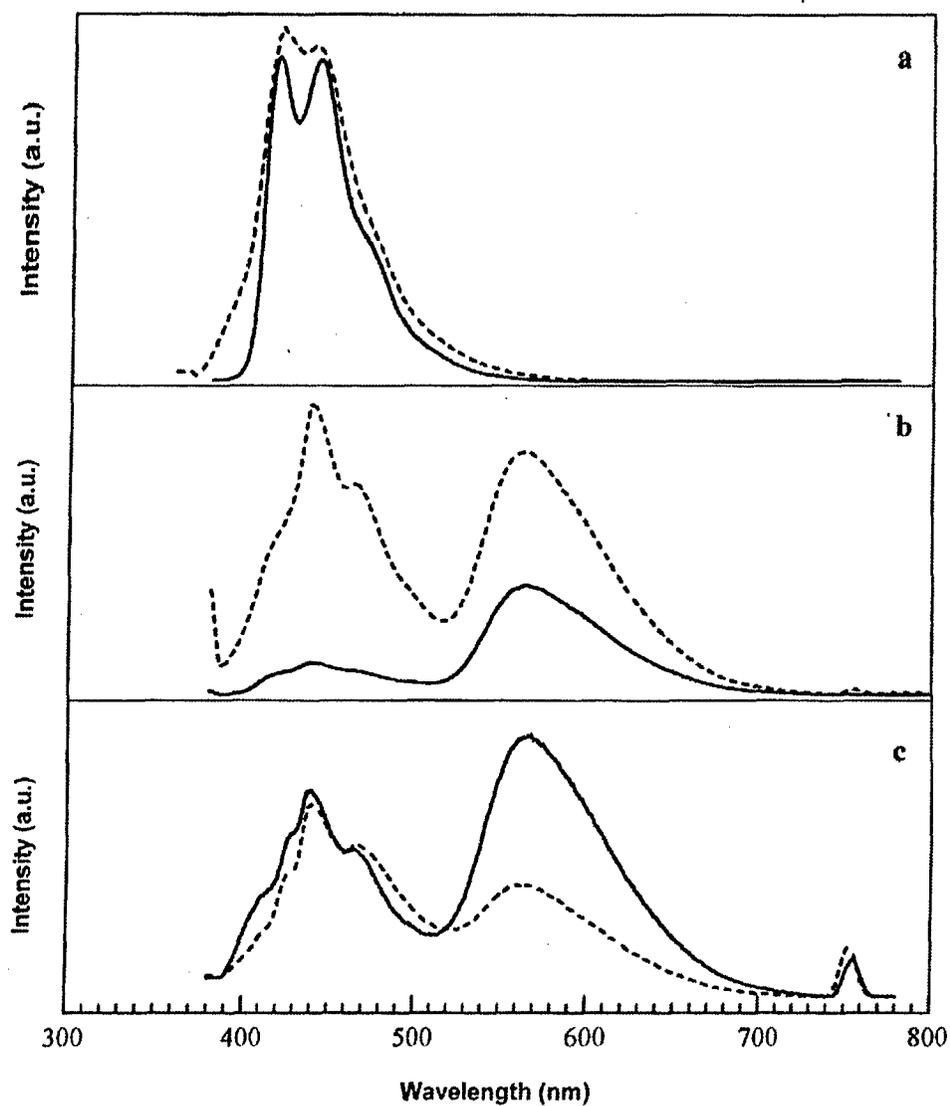


Figure 2: Comparison of solution photoluminescence spectra for **1** (—) and **3** (- - -) in chart (a); for **2** at 10 micromolar (—) and 500 micromolar (- - -) in chart (b), for **4** at 27.6 micromolar (—) and 41.4 micromolar (- - -) in chart (c). All spectra in chloroform at room temperature. The spectra in chart (a) are scaled on the ordinate relative to one another in each chart for ease of comparison of peak maxima; charts (b)-(c) each show spectra using the same ordinate scale.

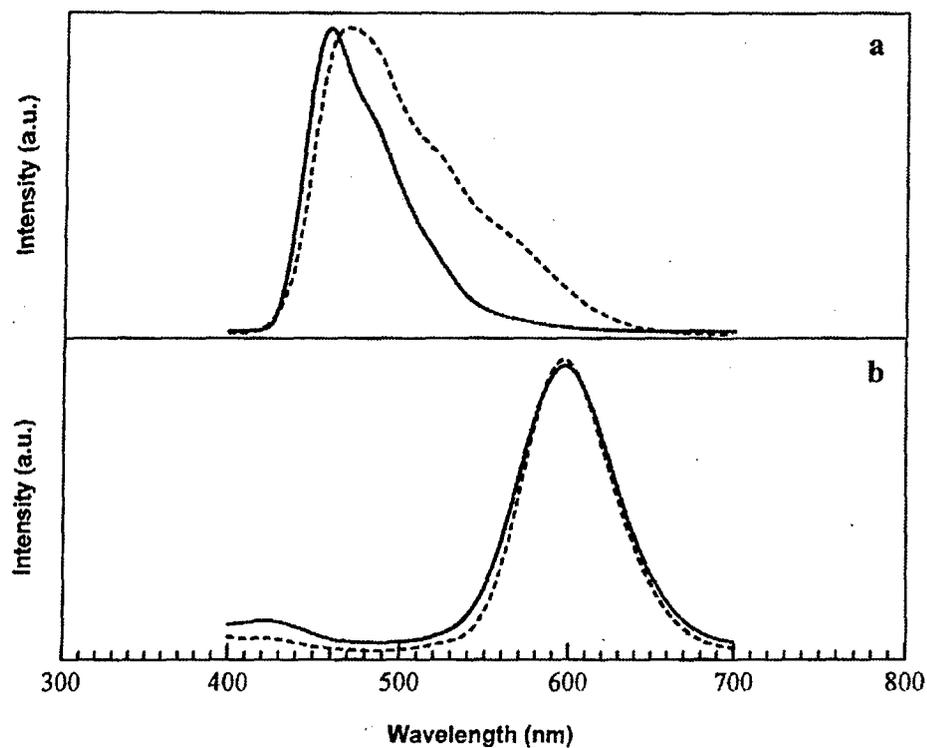


Figure 3: Comparison of electroluminescence spectra for **1** (-) and **3** (---) in chart (a); for **2** (-) and **4** (---) in chart (b). Spectra on each chart are scaled on the ordinate relative to one another in each chart for ease of comparison.

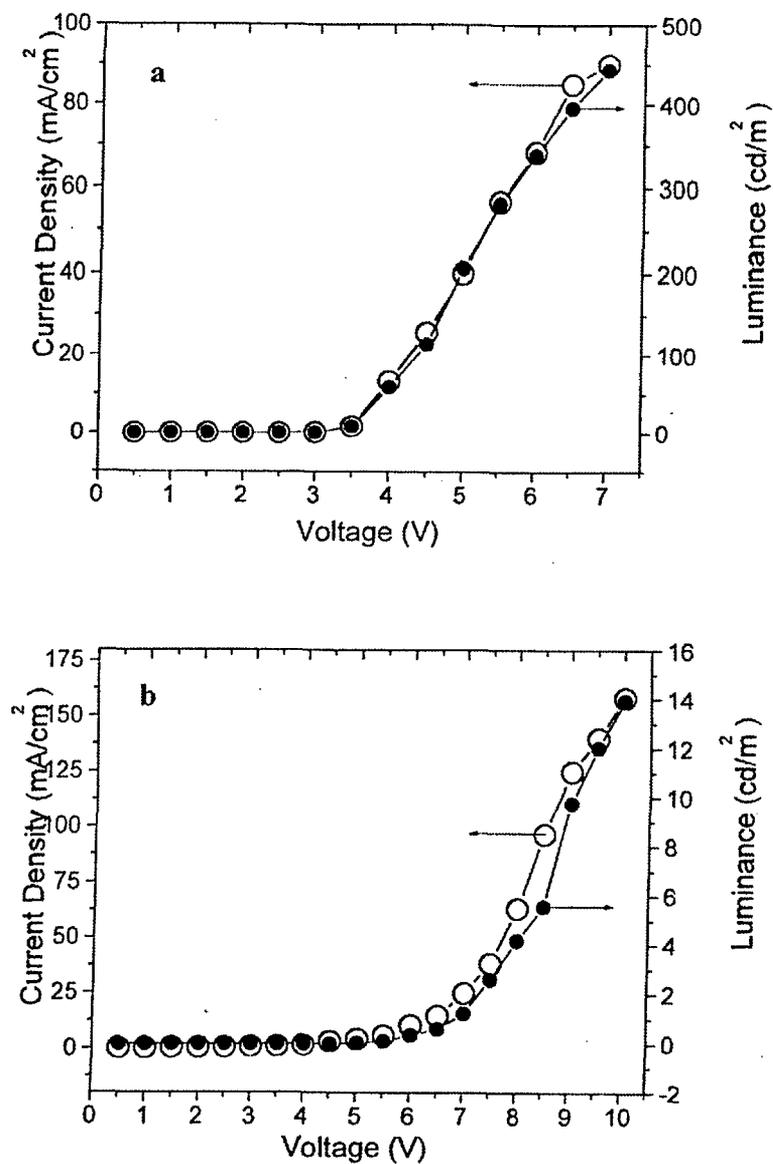
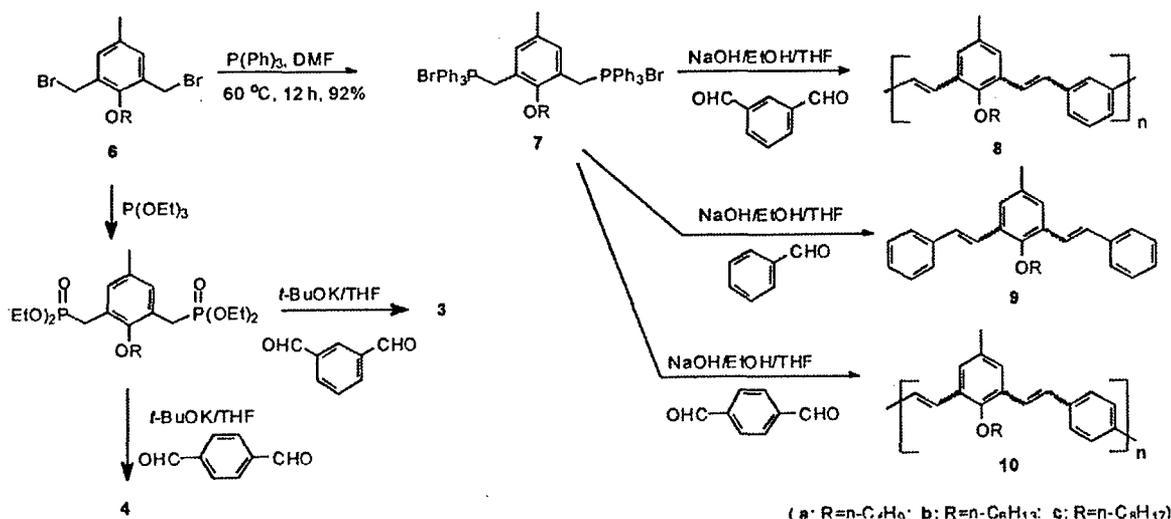


Figure 4: Current-voltage-luminescence plots for ITO/PEDOT:PSS/(emissive layer)/Ca-Al LEDs based upon **1** (chart a) and **3** (chart b).

In another study the effect of *cis/trans* bond ratio on luminescence in PPV type chromophores, poly[(2-alkoxy-5-methyl-1,3-phenylenevinylene)-*alt*-(1,3-phenylenevinylene)s and poly[(2-alkoxy-5-methyl-1,3-phenylenevinylene)-*alt*-(1,4-phenylenevinylene)s have been synthesized by the Wittig reaction to provide 45-62% *cis*-vinylene bonds. Optical characteristics of these compounds were compared with their respective isomers whose *cis*-vinylene contents are significantly lower (9-16%). Although the greater content of *cis*-CH=CH linkages causes the absorption λ_{max} to be slightly blue-shifted (by ~3-6 nm), the impact of the vinyne bond geometry appears to be negligible on their fluorescence spectra. Fluorescence quantum efficiency of these polymers are estimated to be $\phi_{\text{fl}} \approx 0.25$ and 0.72, respectively. In addition, both polymers are strongly electroluminescent, showing effective color tuning via controlled insertion of *m*-phenylene. Thus it is shown that the ratio of *cis-trans*-vinyne bond configuration exerts a large impact on the EL device performance.

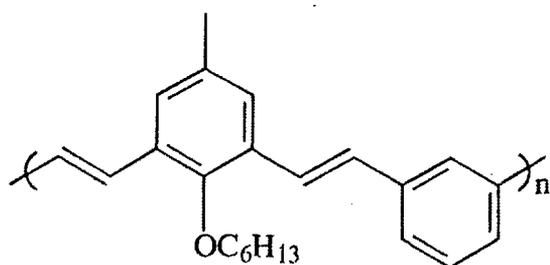
Polymer synthesis using the Wittig reaction.



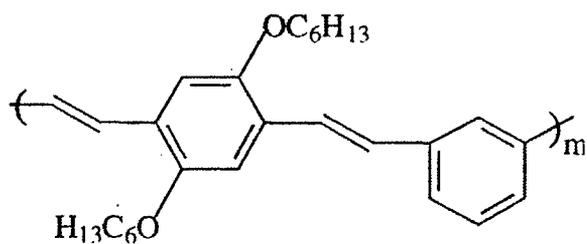
The EL emission λ_{max} values or colors of the polymers are about the same as those of their respective isomers. The external EL quantum efficiency of the 1,3 compound however, is about 20 times lower than its 1,4 isomer, showing a large impact of the *cis/trans*-vinylene ratio. The negative impact of *cis*-vinylene to the EL efficiency diminishes significantly in longer chromophore lengths, as the EL efficiency is only slightly decreased from that of its isomer. Similar trend is observed from the PL and EL quantum efficiencies of both isomer pairs suggesting that *trans*-CH=CH is the desired bond configuration for optimum luminescence efficiency.

In another investigation, the photoluminescence and electroluminescence of violet-blue (I) and green (II) emitting polymers and their blends were studied. This included the effects of excitation wavelength, concentration and film thickness on the photoluminescence; also double-layer LEDs (ITO/PEDOT/polymer/Ca/Al) were fabricated and compared with single-layer LEDs. A strong dependence of the PL spectra on excitation wavelength for the violet-blue polymer in solution was observed. On increasing the applied voltage, the EL spectra show a blue shift, and the emission intensity for the observed higher energy transition increases at the expense of the lower energy transition. Förster energy transfer in the photoluminescence and electroluminescence of the polymer blend was also studied. The results indicate that the energy transfer was independent of excitation wavelength and applied voltage. The LED using the blend was found to be more efficient than either of the devices using polymers I or II, because polymer I provides excitons which migrate to the chain of polymer II and contribute to the total emission.

Furthermore, polymer I acts as a diluent to reduce exciton quenching. The chromophore blend also aids the separation of PL output from absorption, thereby alleviating self-absorption and improving the device efficiency.



Polymer I



Polymer II

Structures for polymers I and II.

Finally, we highlight an optoelectronic study of a series of alkoxy-substituted phenylene ethynylene(PE)/phenylene vinylene(PV) hybrid conjugated polymers which showed that longer alkoxy side chains improve EL efficiency. In particular, polymers with

longer side chains on the PV moiety show this increase. With increasing solvent polarity, both the absorption and emission spectra of the polymer show bathochromic shifts. With increasing chromophore concentration, the solution PL spectra show red shifts and new bands indicating intermolecular excimer formation at the higher concentrations. The 0-0 band of the solution PL spectra is sensitive to excitation energy. When adding nonsolvent methanol to the polymer/chloroform solution, both the absorption and emission spectra show red shifts and new bands, indicating aggregate and excimer formation respectively. Specifically, the new band at 441-428 nm in the PL spectra of the polymer/chloroform/methanol solution indicates aggregate formation.

Further details of these studies are given in the respective publications.

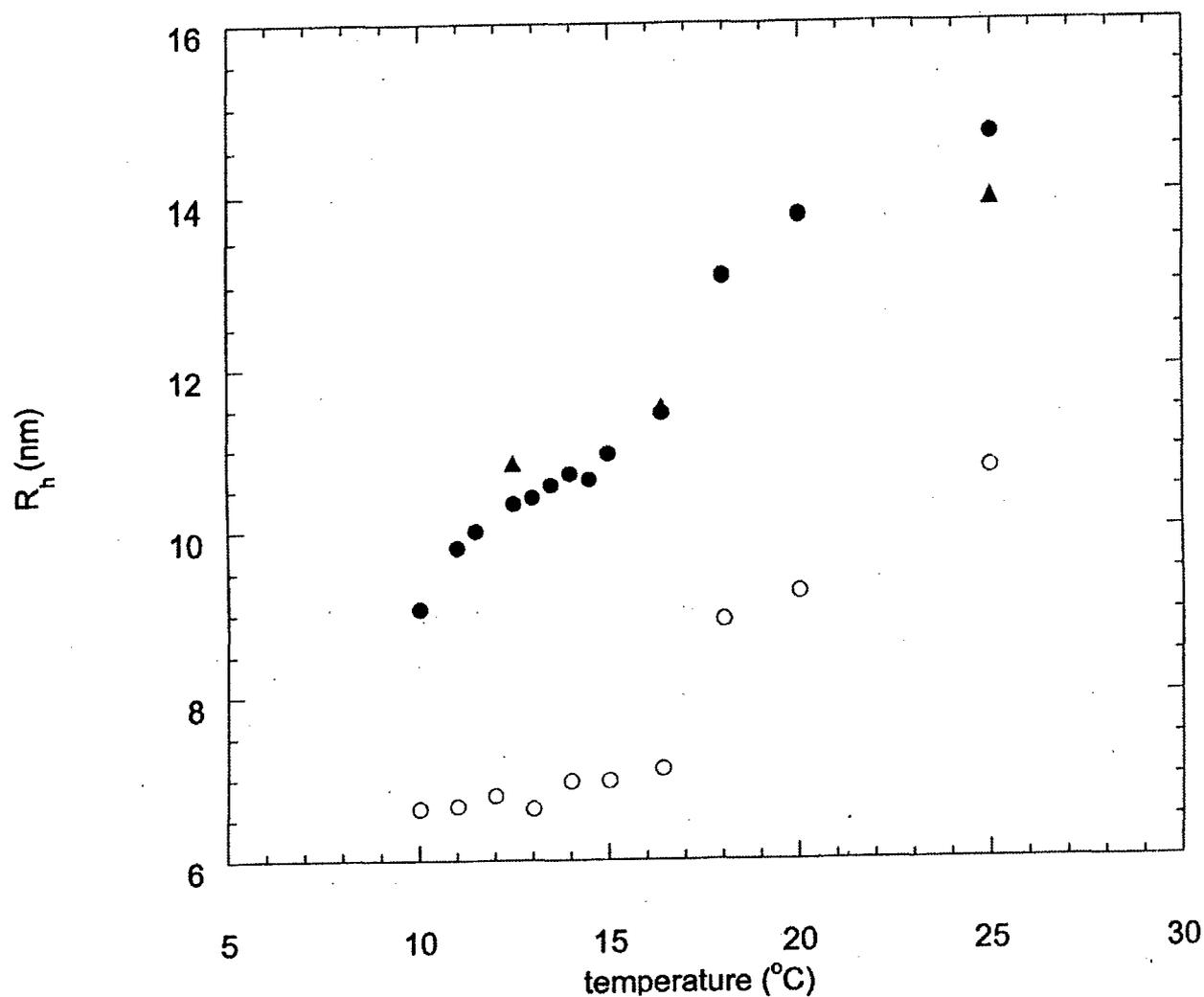
We also proposed new electrode materials for use in PLEDs. Several materials have been studied for suitability as an LED anode, including doped zinc oxide, titanium nitride, niobium diselenide, Zr-doped ZnO, and other transparent conducting oxides. The most extensively used is indium tin oxide (ITO), which has high optical transparency, high conductivity, and provides a high work function substrate for hole injection into a variety of organic materials. Films can be prepared in a variety of ways, such as chemical vapor deposition, pulsed laser deposition, sputtering, spray pyrolysis, and sol-gel techniques. We reported the first use of films produced from ITO nanoparticles as anode electrodes for PLED devices. The properties of these films in LED devices were compared with those of control LEDs fabricated using conventional ITO/glass substrates commercially available. In a selected system, we found a luminance efficiency (0.13 cd/A), higher than that (0.09 cd/A) obtained in control devices fabricated on conventional commercial ITO anodes. The

thin films were made by spin coating of a nanoparticle suspension followed by annealing. The ITO nanoparticle films have a stable sheet resistance of 200 Ω /sq, and an optical transmittance greater than 86% over the range of 400-1000 nm. Their textural property was also studied. The results demonstrate that ITO nanoparticle can form high efficient and reproducible anodes.

B. Solution Properties of Flexible Macromolecules (4,20)

A fundamental phenomenon in polymer science is the coil-globule transition of a single solvated macromolecular chain which occurs when a polymer is quenched from a good solvent into a poor solvent. This transition has been the subject of many investigations. Most coil-globule transition experiments have been carried out using polystyrene solutions of extremely low concentration. Various experimental methods such as static and quasielastic light scattering, small angle neutron scattering, ultracentrifuge and viscometric measurements have been used for the determination of linear polymer dimensions. Contradictory experimental results for the contraction of polystyrene in cyclohexane have been reported in early studies, one yielding a very sharp transition with the other indicating a gradual contraction. This small decrease in size does not satisfy criteria for a densely packed collapsed globule. Our recent work which extended coil-globule studies to highly dilute solutions of macromolecules containing polar groups, such as the halogenated polystyrenes, poly(4-chlorostyrene) and poly(2-chlorostyrene), show that chain contractions can exceed those reported in earlier studies of polystyrene/cyclohexane system, but are not totally consistent with single globule formation.

Since the prediction of the collapse of a polymer chain from a coil to rather dense form, the coil-globule transition has also been subject to extensive theoretical research. We have now studied the coil-globule transition behavior of sodium poly(styrene sulfonate) (Na PSS) ($M_w=1.2 \times 10^6$ g/mol) in 4.17 M aqueous NaCl solution by dynamic light scattering. The polymer dimensions show a smooth and continuous contraction below the reference temperature (18 °C). The hydrodynamic radius ($\langle R_h \rangle$) of NaPSS in this system was reduced from 13.1 nm (18 °C) to 9.1 (nm) at 10 °C. At the latter temperature the polymer segment volume fraction was calculated to be 0.58. The contraction for this and other NaPSS samples investigated in this study is small because under these high ionic strength conditions the polymer chains were contracted before collapse as a result of the screening effect of the ions. The globular density at 10°C is in accord with recent theory.



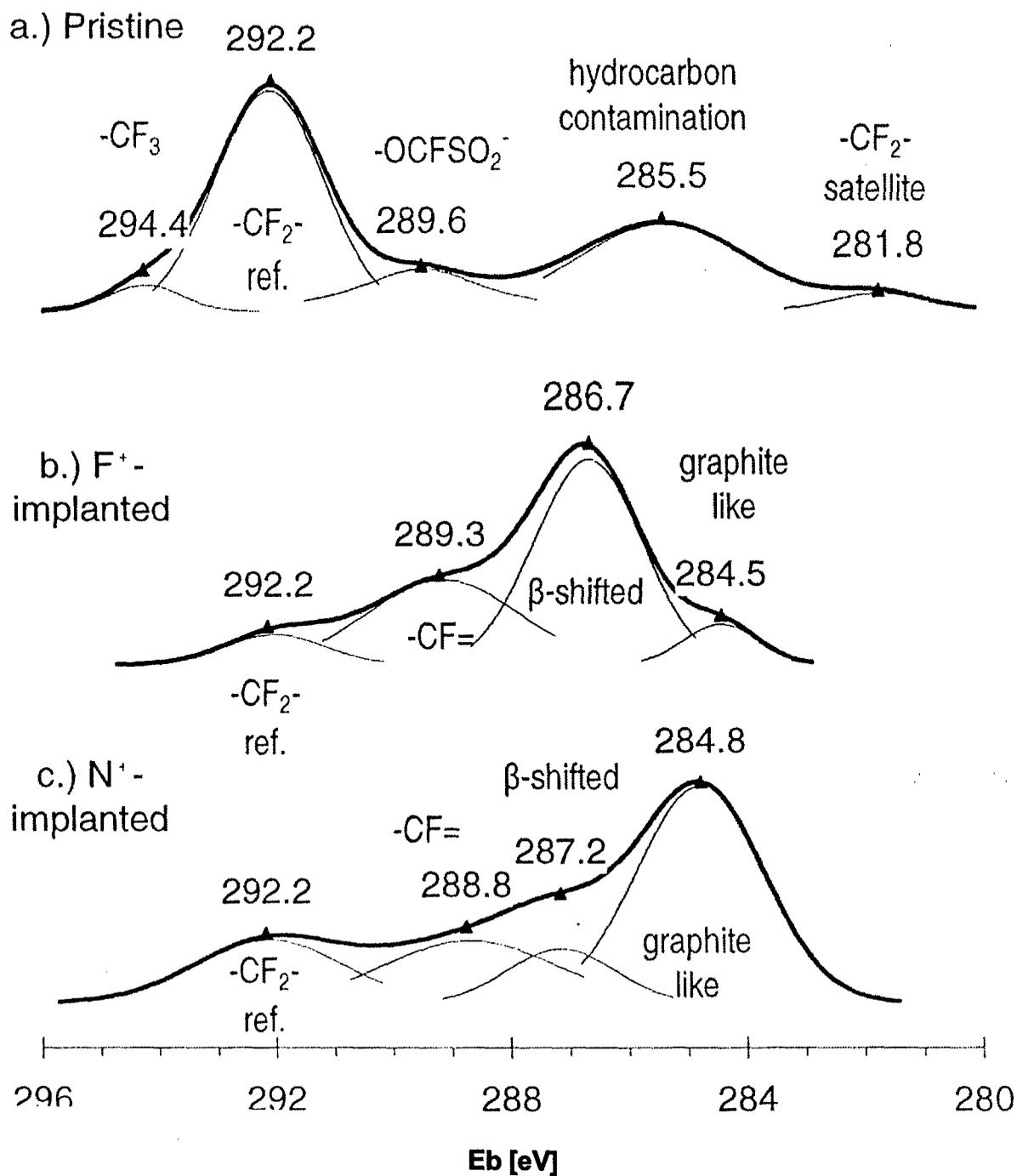
Hydrodynamic radius of two samples of sodium polystyrene sulfonate as function of temperature

C. Ion Implantation of Polymer Films (19, 25, 35)

The properties of Nafion® surfaces can be modified by ion implantation with N^+ and F^+ . We have carried out an X-ray photoelectron spectroscopic study of the implanted surface. To interpret the XPS Spectra, calculations using a semiempirical quantum chemical formalism AM1 has been applied, in conjunction with the charge potential model,

to predict C_{1s} core electron binding energies.

The calculations with the charge potential model correlate well C_{1s} core electron binding energies for a wide variety of polymers. The theoretical calculations provide a generally useful methodology for interpreting XPS spectra. It was found that ion implantation of Nafion® produces a carbonized surface layer. Analyses of XPS spectra provided a consistent assignment of the spectral components associated with the ion implanted product.



XPS Spectra (C_{1s}) of Pristine and Ion Implanted Nafion®

D. Other Studies (23)

The phase behavior of polymer blends in which one or more constituents are statistical copolymers has been studied with AFOSR support for some time. The presence of a copolymer can create a miscibility window in the case of binary systems, and more complex behavior in ternary and higher systems. In ternary systems the existence, location, shapes and size of such windows is highly sensitive to constituent parameters and thereby offers opportunities for more accurate determination of segmental interaction parameters. Thus we have studied the phase behavior of ternary blends of poly(2,6-dimethyl-1,4-phenyleneoxide) (PPO), polystyrene (PS) and a 50/50 mole % statistical copolymer of o-chlorostyrene and p-chlorostyrene [p(o-CIS-p-CIS)] and analyzed this in terms of a Flory-Huggins mean-field treatment. Both PS/PPO and PPO/p(oCIS-pCIS) binary blends exhibit single glass transition temperatures over the full composition range whereas the PS/p(oCIS-pCIS) system displays a substantial immiscibility window which extends into the ternary phase diagram.

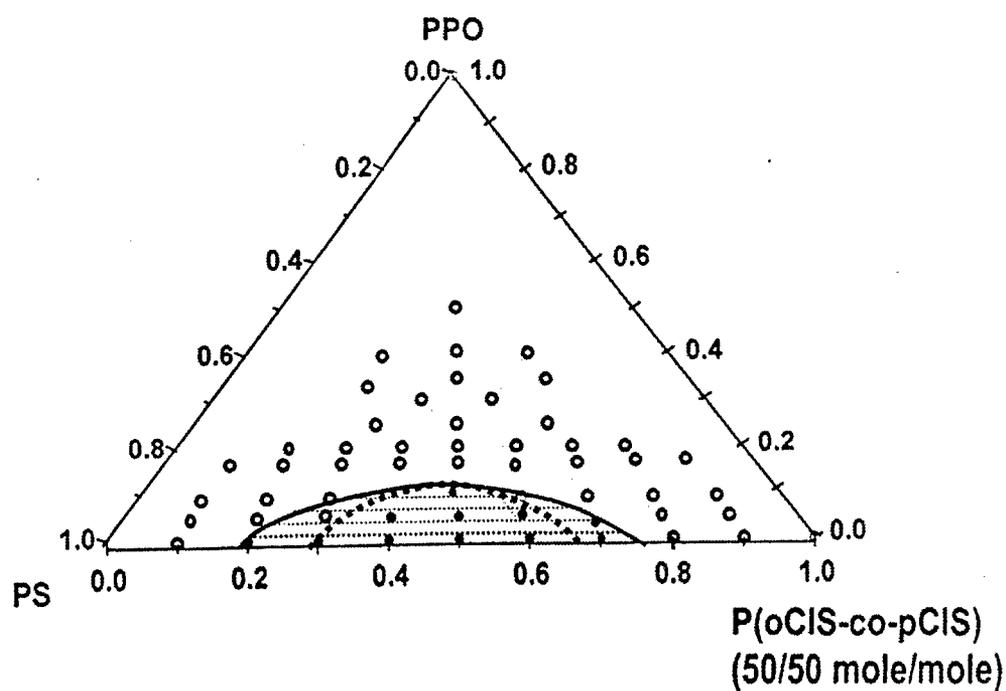


Figure 6. Phase diagrams for blends of PS/PPO/[p(oCIS-pCIS)] (----) bimodal boundary, (.....) Spinodal boundary, o miscible blend, • immiscible blend

A typical ternary phase diagram sharing both bimodal and spinodal boundaries is shown in the figure. Several molecular weights of PS were studied; the boundary showed high sensitivity to this parameter. As a result a consistent set of the respective segmental interaction parameters of considerable accuracy were obtained.

VIII. PUBLICATIONS

Forty scientific papers (36 published, 4 in press) were completed with full or partial AFOSR support during the grant period.

1. Synthetic Metals, 130, 203-212 (2002) (with S-H Jin, H-J Lee, Y-S Gai, T. Zyung, H-N Cho and E.E. Gürel) "An Alkylsilylphenoxy PPV Chromophore"
2. Thin Solid Films, 417, 147-150 (2002) (with E.E. Gürel and Y. Pang) "Luminescence Properties of Modified Poly(*m*-phenylenevinylene)-*alt*-*p*-phenylenevinylene): Effects of Side-chain Length, Blending and Device Configuration"
3. Macromolecules, 35, 7569-7574 (2002) (with Q. Chu, Y. Pang and L. Ding) "Synthesis, Chain Rigidity and Luminescent Properties of Poly[(1,3-phenyleneethynylene)-*alt*-tris(2,5-dialkoxy-1,4-phenyleneethynylene)]s"
4. Polymer, 43, 5439-5445 (2002) (with E. Serhatli, M. Serhatli and B.M. Baysal) "Coil-Globule Transition Studies of Sodium Poly(Styrene Sulfonate) by Dynamic Light Scattering"
5. Macromolecules, 35, 5720-5723 (2002) (with L. Liao and Y. Pang) "Comparison of Optical Properties Between Blue-Emitting Poly(*m*-phenylenevinylene) and PPV Block Copolymer"
6. Euro-Mediterranean Post-Graduate Advanced School: Lectures (E. Martuscelli and L. D'Orazio, Eds.) Naples, 107-112 (2002) (with E. Gürel) "Newer Techniques for Fibre Characterization".
7. Macromolecules, 35, 9939-9946 (2002) (with M. Zheng, L. Ding and Z. Lin) "Synthesis and Characterization of Fluorenediylvinylene and Thiophenediylvinylene-Containing Terphenylene-Based Copolymers".
8. Synthetic Metals, 132, 227-234 (2003) (with A. Sarker, E. Gürel, L. Ding, E. Styche and P.M. Lahti) "Light Emitting Poly(*para*-phenylenevinylene-*alt*-3-*tert* butyl-*meta*-phenylenevinylene)s".
9. J. Appl. Phys., 93, 1995-2001 (2003) (with B. Hu) "Blue, Green, Red and White Electroluminescence from Multichromophore Polymer Blends".
10. Materials Research to Meet 21st Century Defense Needs, Chapter 6, "Functional Organic and Hybrid Materials", National Academic Press, Washington, D.C., 2003

11. Macromolecules, 36, 3848-3853 (2003) (with Q. Chu, Y. Pang and L. Ding) "Green-Emitting PPE-PPV Hybrid Polymers: Efficient Energy Transfer across the *m*-Phenylene Bridge".
12. J. Photosci., 10, 149-155 (2003) (with M. Aguiar, L. Akcelrud, M. Pinto, T. Atvars, and J. Saltiel) "Spectroscopic Evidence for Aggregation of Stilbene Derivatives in Solution".
13. J. Polym. Sci.: Part A, 41, 2650-2658 (2003) (with L. Liao, Y. Pang and L. Ding) "Blue-Emitting Poly[*m*-phenylene vinylene)-*alt*-(*o*-phenylene vinylene)]s: The Effect of Regioregularity on the Optical Properties".
14. J. Phys. Chem. A, 107, 6533-6537 (2003) (with A.M. Sarker, U. Kaneko and P.M. Lahti) "Excited States of Bromine-Substituted Distyrylbenzenes: Models for Conjugated Polymer Emission".
15. Macromolecules, 36, 7301-7307 (2003) (with L. Ding, Y. Lin, Y. Pang and L. Liao) "Photoluminescence and Electroluminescence Study of Violet-Blue and Green Emitting Polymers and Their Blend".
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