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DESCRIPTION

CROSS-REFERENCE TO RELATED APPLICATION

[0001] The present application claims the benefit of U.S. Provisional Application Serial No. 61/000,908, filed October 30, 2007, which is hereby incorporated by reference herein in its entirety, including any figures, tables, or drawings.

BACKGROUND OF THE INVENTION

[0002] Polymeric electrochromics capable of a fast and reversible color change upon electrochemical oxidation and reduction have received a considerable attention over the past decade. A particular emphasis has been placed on incorporating the most stable of these electroactive materials in devices such as windows, mirrors (rear-view/side-view mirrors for cars) and displays, for anticipated industrial and commercial applications. WO 03/046106 provides an example of such an electroactive material, which is a conducting polymer based on 3,4-alkylenedioxyppyrrrole and 3,4-alkylenedioxythiophene. While a number of neutral state red and blue absorbing conjugated polymers have been synthesized and integrated into electrochromic devices, attempts at synthesizing saturated green polymers chemically or electrochemically, which can switch to a transmissive state, have met with limited success due to the complex nature of the required absorption spectrum that must contain at least two bands in the neutral state of the material.

[0003] To date, only one article reports the existence of a green conjugated polymer with a transmissive state (with a blue hue) upon oxidation. (Durmus *et al.*, *Chem. Commun.*, 2007, 3246-3248). However, this material is prepared by an electrochemical polymerization/film deposition and does not show any solubility/processability making it difficult to be integrated into devices, restricting the scope of possible applications. Accordingly, soluble neutral state green conjugated polymers with high electrochromic contrasts, fast switching times and highly transmissive oxidized states would be a desirable improvement in the field of conjugated polymers. Additionally, materials that are solution processable would provide advantages in a wide range of applications.

BRIEF SUMMARY OF THE INVENTION

[0004] This invention pertains to a method for forming a conjugated polymer according to Claim 1.

[0005] It also pertains to the conjugated polymer thus obtained, as defined in Claim 2. The conjugated polymer is constructed with a plurality of repeating units where the repeating units have two specific substituted dioxyheterocycle based donor groups coupled to an acceptor group. The conjugated polymer absorbs radiation within a first band of the visible spectrum and a second band of the visible spectrum when in a neutral state but is transmissive in these bands in an oxidized state. The absorbance of radiation within the first and second bands in the oxidized state is less than in the neutral state to the extent, often by 200% or more, such that one can discern the polymer as colored in the neutral state but of little or no color in the oxidized state. The first absorption band can have a visible absorption maximum at a wavelength below about 500 nm and the second absorption band can have a visible absorption maximum at a wavelength above about 550 nm and a local minimum between 480 and 580 such that the neutral state colored polymer is green in appearance.

[0006] The polymer can be soluble in one or more solvents, for example methylene chloride, chloroform, tetrachloroethane, tetrahydrofuran, dioxane, benzene, toluene, xylenes, chlorobenzene, dichlorobenzene, pyridine, ethyl acetate, butanol, ethanol, methanol, acetonitrile, acetone, isopropanol, water and mixtures thereof. The polymer can then be prepared and processed into films or coatings from solution. The dioxyheterocycle of the polymer is a 3,4-propylenedioxythiophene. The acceptor group is an electron poor aromatic unit selected from specific substituted or unsubstituted benzothiadiazole groups,

[0007] The method of forming a conjugated polymer according to the invention includes steps of reacting two donor compounds having a specific substituted dioxyheterocyclic moiety with a specific conjugated acceptor compound having an acceptor moiety to form a polymerizable unit, which is an oligomer where dioxyheterocycle based donor groups are attached to an acceptor group. A plurality of these oligomeric polymerizable units can then be linked covalently to form the conjugated polymer, where the conjugated polymer absorbs radiation within a first band of the visible spectrum and a second band of the visible spectrum when in a neutral state and upon oxidation the polymer is transmissive as described above. The polymer is soluble in at least one

solvent. The dioxyheterocyclic moiety has a coupling moiety which is an organotin substituent, and the conjugated acceptor compound contains a pair of complementary halogen functional groups symmetrically situated on the conjugated acceptor compound.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008]

Figure 1 shows absorbance spectra of polymers (a) **6a**, (b) **6b**, (c) **9** and (d) **12** respectively where thin films were cast onto ITO from toluene (**6a**, **6b**, **9**) or chloroform (**12**) and electrochemically oxidized in a 0.1M solution of LiBF₄ in Acetonitrile.

Figure 2 shows relative luminance measured on thin films of Polymers **6a** (P([ProDOT-OOct₂]₂-BTD)) (●), **6b** (P([ProDOT-OEtHex₂]₂-BTD)) (▲), **9** (P([ProDOT-OOct₂-Th]₂-BTD)) (■) and **12** (P([ProDOT-OOct₂-Th]₂-BTD)) (▼). Experiments were carried out using solid thin films spray cast onto ITO from toluene (**6a**, **6b**, **9**) or chloroform (**12**).

Figure 3 shows photographs of the neutral and oxidized polymers (a) **6a** (P([ProDOT-OOct₂]₂-BTD)), (b) **6b** (P([ProDOT-OEtHex₂]₂-BTD)), (c) **9** (P([ProDOT-OOct₂-Th]₂-BTD)) and (d) **12** (P([ProDOT-OOct₂-EDOT]₂-BTD)) as solid thin or/and thick films spray cast onto ITO from Toluene (**6a**, **6b**, **9**) or Chloroform (**12**).

DETAILED DESCRIPTION OF THE INVENTION

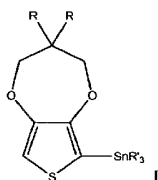
[0009] One embodiment of the invention is a new and efficient route towards synthesizing novel, soluble conjugated polymers that are green in the neutral state and high transmissive in the oxidized state. According to an embodiment of the invention, the soluble conjugated polymer provides one or more of the following advantages: high optical contrast in the visible region; outstanding electrochemical switching properties; charge transport properties, as desired for green colored solar cells; and the ability to be processed into electrochromic devices using convenient deposition methods, such as spin coating, roll-to-roll coating, spray casting, and various methods of printing (e.g. ink jet printing).

[0010] To achieve a neutral state green polymer, absorption in the red and the blue regions of the visible spectrum is required. Fine tuning of the energy gap, as well as introduction of an adequate set of absorption bands in the electromagnetic spectrum, is achieved by the choice of the structure of the repeating units that compose the conjugated portion of the polymer. Structural parameters are controlled to achieve the desired optical properties and processability for this new family of soluble polymers. Careful analysis and comparison of the bathochromic shift occurring upon polymerization can allow determination of an expected absorption spectrum of a single polymerizable unit to be synthesized and polymerized. This polymerizable unit comprises electron donating and accepting moieties that constitute the backbone of the electrochromic polymer.

[0011] In this invention, a dioxythiophene moiety having solubilizing chains substituted thereon comprise the donating moieties within the polymerizable units employed to form the polymers. The solubilizing chains are alkoxy solubilizing chains (linear or branched), which allow processing into solid thin film by solution casting and can enhance transmissivity of the oxidized state. For a polymeric material transmitting/reflecting green light, obtaining highly transmissive contrasts upon oxidation requires simultaneous and efficient bleaching of the two absorption bands that occur in the blue and red portions of the visible spectrum.

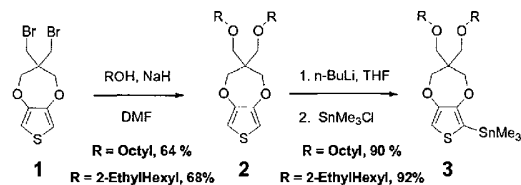
[0012] The soluble conjugated polymers of the invention are prepared by coupling dioxyheterocyclic donor moieties with acceptor moieties via coupling reactions. Coupling reactions that can be used, include, but are not restricted to: Stille coupling, Kumada coupling, Hiyama coupling, Negishi coupling and Suzuki coupling.

[0013] In one embodiment of the invention Stille coupling is employed as the coupling reaction. In this embodiment, the polymerizable units are prepared using stannylated dioxythiophenes, which can be prepared using any suitable technique, followed by an oxidative polymerization of the units. The units are appropriately functionalized oligomers of donor and acceptor moieties that in combination yield the electrochromic properties of the green to transmissive polymers. The stannylated dioxythiophene compound is 3,4-propylenedioxythiophene of formula 3 in Scheme 1, below, shown as trimethyl tin (-SnMe₃)A general formula for a stannylated 3,4-propylenedioxythiophene is given in Formula I:



where R groups are as defined in Claim 1.

Scheme 1



[0014] An embodiment of the invention includes preparing stannylated alkylenedioxythiophenes, as shown in Scheme 1, above, where a monostannylated bis-alkoxymethyl substituted 3,4-propylenedioxythiophene (**3**), which can be coupled into a polymerizable unit. The synthesis begins with a facile nucleophilic substitution of alkoxy derivatives (solubilizing group) for Br of compound **1**, which is shown as a Williamson etherification in *N,N*-dimethylformamide (DMF) where a sodium alkoxide is formed by the reaction of an alcohol with sodium hydride to form compound **2**. The R groups of scheme 1 are shown as being octyl or 2-ethylhexyl, although any suitable linear or branched alkyl groups, such as C2 to C30 alkyl groups, can be employed. Appropriate reaction conditions for etherification reactions are known to those skilled in the art. Other reactions can be employed to form appropriately substituted 3,4-propylenedioxythiophenes for use as donor moieties to form the polymerizable units.

[0015] The resulting symmetrical alkoxy substituted compound **2** of Scheme 1 is monolithiated using a suitable organolithium reagent, such as, for example, *n*-butyllithium, *t*-butyllithium, or methylolithium. Other sufficiently basic reagents can be employed for the preparation of an organometallic intermediate, where the metal can be, for example, any alkali or earth alkali metal, for the preparation of the monostannylated product **3**. The reaction can be performed in any suitable organic solvent, generally a polar aprotic solvent such as tetrahydrofuran (THF). The resulting compound is then monostannylated by replacing the resulting lithium substituent with an organotin substituent using any suitable tin containing reagent, such as trimethyltin chloride or tributyltin chloride, to afford the relatively air-stable compound **3** in high yield.

[0016] The stannylated alkylenedioxythiophenes can then be used to form oligomers having alkylenedioxythiophene end groups, which are the polymerizable units. This process includes mixing the stannylated alkylenedioxythiophenes with an additional compound, which contains at least one acceptor moiety. Compound **3** thus reacts with one or more halogenated compounds containing the acceptor moiety.

[0017] Scheme 2 illustrates an example of a method according to an embodiment of the invention that progresses through the construction of soluble and symmetric conjugated oligomers, polymerizable units, having donor and acceptor moieties and through the polymerization of the oligomers to yield linear conjugated polymers. Table 1 discloses the characterization of a number of polymers according to the invention. As illustrated in Scheme 2, a Stille reaction can be used to couple stannylated compound **3** with dibrominated acceptor containing species **4**, **7** or **10**. Other halogens, such as chlorine, iodine, or even fluorine can be used in place of the bromine on the acceptor compounds. The acceptors are generally, but not necessarily, symmetrically halogenated where halogens are positioned on equivalent opposite sites of the acceptor compounds, as shown for compounds **4**, **7** and **10**. Stille reaction processes are well known in the art, and can be carried out using, for example, a Palladium(0) or Palladium(II) catalyst. In many embodiments, the reaction is carried out under air and moisture-free conditions to maximize the reaction yields. Soluble oligomers **5**, **8** and **11** can be isolated by various techniques, including, for example, column chromatography using silica as the stationary phase and varying the ratios of solvents employed for elution. Such solvents can include, for example, mixtures of hexanes, dichloromethane and ethyl acetate.

[0018] The oligomers can be polymerized in a step-growth fashion where the alkylenedioxythiophene end groups self-condense to form the conjugated polymer. For example, as illustrated in Scheme 2, the target oligomers **5**, **8** and **11** can be self-condensed in chloroform, or other suitable solvent, through the low oxidation potential 3,4-propylenedioxythiophene chain-ends using ferric chloride as an efficient oxidative agent to afford fully conjugated and well-defined electroactive polymers **6a**, **6b**, **9** and **12** as indicated in Table 1. Polymer purification can be carried out by precipitation in MeOH, reduction in chloroform using hydrazine monohydrate followed by a 48 hours Soxhlet purification. (see Table 1) Although the "R" groups of scheme 2 are shown as being octyl or 2-ethylhexyl, any suitable linear or branched alkyl groups, such as C2 to C30 alkyl groups, can be employed to enhance

processability, fine tune the color, including the hue and saturation of the green color, enhance transmissivity of the oxidized form, and modify the solid film's morphology.

Scheme 2

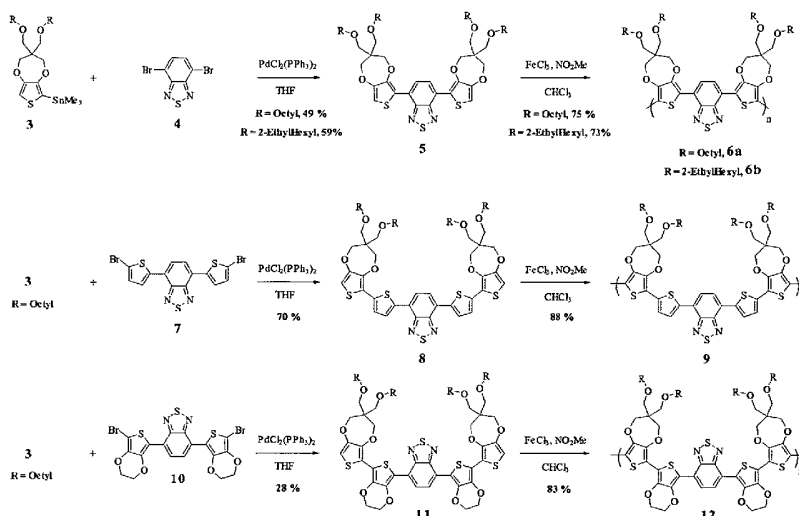


Table 1. GPC Estimated Molecular Weights in THF, Isolated Yields for the Polymerizations, Elemental Analysis of the Copolymers **6a** (P([ProDOT-OOct₂]₂-BTD)), **6b** (P([ProDOT-OEtHex₂]₂-BTD)), **9** (P([ProDOT-OOct₂-Th]₂-BTD)) and **12** (P([ProDOT-OOct₂-EDOT]₂-BTD)).

Polymer	M_n (g/mol)	M_w (g/mol)	PDI	Avg. no. of repeat units	Avg. no. of rings	Yield (%)	EA (Calcd/Found)		
							C	H	N
P([ProDOT-OOct ₂] ₂ -BTD)	14700	32150	2.2	15	45	75	66.5/66.42	8.57/8.64	2.77/2.79
P([ProDOT-OEtHex ₂] ₂ -BTD)	27800	59000	2.1	27	81	73	66.5/66.21	8.57/8.48	2.77/2.73
P([ProDOT-OOct ₂ -Th] ₂ -BTD)	18900	60300	3.2	16	80	88	65.38/66.08	7.72/7.75	2.38/2.21
P([ProDOT-OOct ₂ -EDOT] ₂ -BTD)	10300	17800	1.7	8	40	83	63.22/62.88	7.33/7.27	2.17/2.28

[0019] The chemistry outlined in Schemes 1-2 illustrate how the optical properties can be controlled by the nature of the conjugated backbone by varying the acceptors.

[0020] In the above exemplary embodiments, the dioxiheterocyclic donor moieties are included in activated coupling reagents that react with halogenated compounds including acceptor moieties. In alternative embodiments, the acceptor moieties can be part of the activated coupling reagents and the dioxiheterocyclic donor compounds can have halide functionality for coupling into polymerizable units. For example, instead of being halogenated, the acceptor compounds described above can include a coupling group selected from the group consisting of organotin substituents (including any of the organotin groups described above), organoboron substituents, organomagnesium substituents, organozinc substituents, and organosilane substituents. Such coupling groups can be positioned, for example, to replace the two halogen functional groups of the above illustrated acceptor compounds. This acceptor compound can be coupled with a dioxiheterocycle based donor compounds, as described above, where a monohalogen group allows coupling to the difunctionalized acceptor compound described above.

[0021] The resulting polymers formed from the above described methods comprise a plurality of repeating units, which comprise at least two substituted dioxiheterocycle based donor group coupled to an acceptor group.

[0022] The number average molecular weight of the conjugated polymers of the present application can vary widely, depending on the particular repeating units employed, and the processing parameters used to form the polymers. Exemplary number average molecular weights can range from about 3000 g/mol to about 100,000 g/mol, as measured by gel permeation

chromatography, although polymers having number average molecular weights outside of these ranges can also be formed.

MATERIALS AND METHODS

[0023] Figure 1 characterizes the optical properties of polymers that were synthesized (**6a**, **6b**, **9**, **12**) and their electroactivity upon electrochemical oxidation. As expected for a neutral state green polymer, all materials characterized showed two absorption bands in the visible region of the electromagnetic spectrum, including a first, moderately high absorption peak in the blue portion (below about 500 nm) and a second absorption peak that is generally higher than the first peak (indicating intense absorption) in the orange to red portion (above about 580 nm), with very little or no overlap in the green region (about 480 to about 580nm), so that a minimum absorption point occurs between the first and second peaks in the green region. For all materials characterized, these two absorption bands bleached simultaneously upon oxidation and a new absorption band arises in the near infrared as charge carriers are formed (polarons and bi-polarons). In embodiments, the intense bleaching of the two π - π^* transitions observed, as well as the quasi-absence of residual tail of the near infrared absorption bands into the visible, are observed and the polymer has a high transmissivity of the oxidized state. Taking the onset of absorption of the most intense absorption band as a reference for calculation, the optical energy gaps of these materials were found in the range 1.43 - 1.54 eV, and can therefore be considered to be "Narrow Band-Gap" polymers.

[0024] Figure 2 shows the relative luminance measured under constant illumination from thin films of polymer that were spray cast onto ITO and submitted to progressive electrochemical oxidation. An average of 30% of optical change upon oxidation characterizes this family of polymers. While the relative luminance of polymer **12** reaches the moderate value of 73%, polymer **6b** shows outstanding transmissivity upon oxidation with a relative luminance value reaching 84% in its fully oxidized state. The photographs reproduced in Figure 3 illustrate this difference where all materials have been photographed in their neutral state (left) and in their fully oxidized state (right) as thin films (top) and/or thicker films (bottom). While polymers **6a** and **6b** reflect a rather "aquamarine" (blue-green or persian green) color in their neutral state, polymer **9** can be considered "pine green". The more red-shifted polymer **12** seems to offer a green certainly more "olive-like" (forest green) seemingly due to a residual dark-yellow reflection. Table 2 summarizes the color coordinates of all synthesized materials as well as the potentials at which the materials undergo optical changes. Examples of neutral state green colors that can be achieved using the conjugated polymers of the present application can include the range of colors defined by the CIE Lab color system having "L" values ranging from about 71 to about 80; "a" values ranging from about -15 to about -23; and "b" values ranging from about -11 to about 14, although green colors outside of this range can also be achieved.

Table 2. Colorimetric Results for the Neutral and Oxidized Polymers **6a** (P([ProDOT-OOct₂]₂-BTD)), **6b** (P([ProDOT-OEtHex₂]₂-BTD)), **9** (P([ProDOT-OOct₂-Th]₂-BTD)) and **12** (P([ProDOT-OOct₂-EDOT]₂-BTD)).

Polymer Film	Charge State	E (V)	L	a	b	Observed Color
P([ProDOT-OOct ₂] ₂ -BTD)	N	0	80	-22	-10	Persian Green
	O	0.8	93	-2	0	Transmissive-Grey
P([ProDOT-OEtHex ₂] ₂ -BTD)	N	-0.05	80	-23	-11	Persian Green
	O	0.7	94	-1	0	Transmissive-Grey
P([ProDOT-OOct ₂ -Th] ₂ -BTD)	N	-0.35	75	-15	1	Pine Green
	O	0.75	91	-3	-3	Transmissive-Clear Blue
P([ProDOT-OOct ₂ -EDOT] ₂ -BTD)	N	-0.6	71	-19	14	Forest Green
	O	0.8	89	-5	-5	Transmissive-Clear Blue

[0025] The conjugated polymers of the present disclosure can be employed in a variety of electronic devices. Suitable examples of such devices include electrochromic windows, mirrors and displays; SolarTurf, or artificial turfs that can harvest solar energy and generate electricity; Common Photovoltaic Devices; Electronic paper; Anti-Stat Conductors and Transparent Conductors; and Field Effect Transistors, supercapacitors, batteries, and other electronic components.

[0026] Other embodiments of the invention will be apparent to those skilled in the art from consideration of the specification and

practice of the invention disclosed herein. It is intended that the specification and examples be considered as exemplary only, with the invention being indicated by the following claims.

REFERENCES CITED IN THE DESCRIPTION

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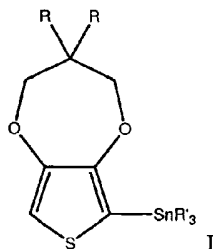
Patent documents cited in the description

- US61000908A [0001]
- WO03046106A [0002]

Patentkrav

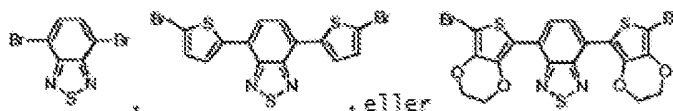
1. Fremgangsmåde til at danne en konjugeret polymer, hvilken fremgangsmåde omfatter trinnene:

5 at reagere to donorforbindelser der har en formel I:



hvor R-grupper er -R''OR''' grupper, hvor R'' er methylen og R''' er en linær eller forgrenet C2- til C30-alkyl og R' er methyl med en konjugeret acceptor-forbindelse omfattende en acceptor-del, hvor den konjugerede acceptor-forbindelse omfattende en acceptor-del er

10



for at danne en polymerbar enhed; og

at forbinde kovalent en flerhed af de polymerbare enheder ved ender af de polymerbare enheder for at danne den konjugerede polymer, hvor den konjugerede polymer absorberer stråling inden i et første bånd af det synlige spektrum og et andet bånd af det synlige spektrum og er grøn i den neutrale tilstand og hvor polymeren ved oxidering er transmissiv med absorbans af stråling inden i det første og andet bånd som er mindre end i den neutrale tilstand, og hvor polymeren er opløselig i mindst et solvent.

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2. Konjugeret polymer fremstillet med fremgangsmåden ifølge krav 1, hvor den konjugerede polymer absorberer stråling inden i et første bånd af det synlige spektrum og et andet bånd af det synlige spektrum og er grøn i den neutrale tilstand og hvor den konjugerede polymer ved oxidering er i en transmissiv tilstand med absorbans af stråling inden i det første og andet bånd som er mindre end i den neutrale tilstand.

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3. Fremgangsmåden ifølge krav 1, hvor R''' er octyl eller 2-ethylhexyl.

DRAWINGS

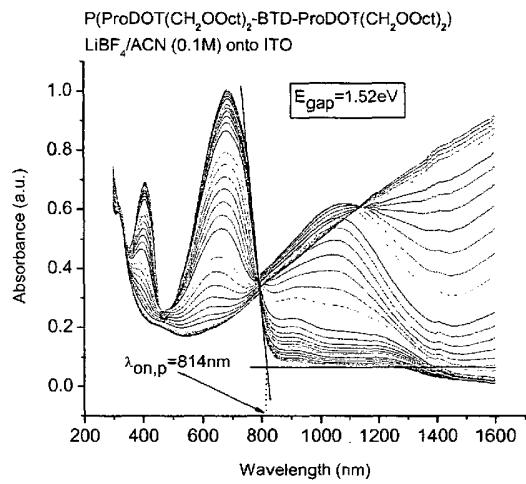


Figure 1A

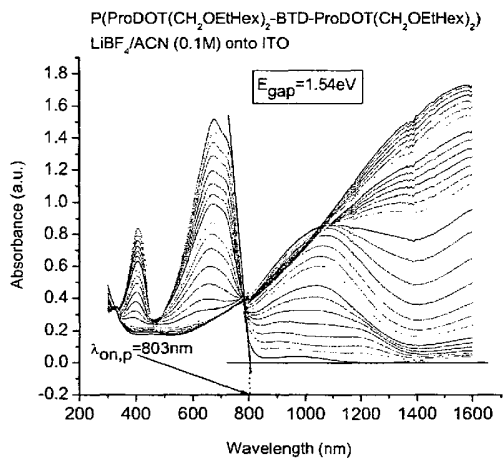


Figure 1B

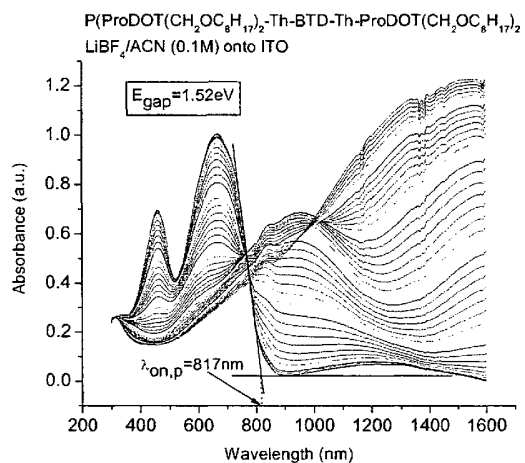


Figure 1C

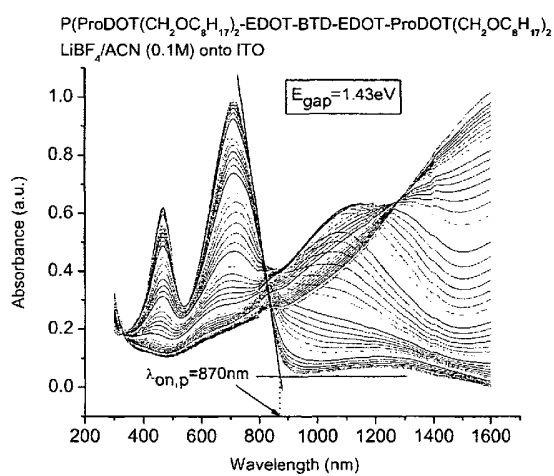


Figure 1D

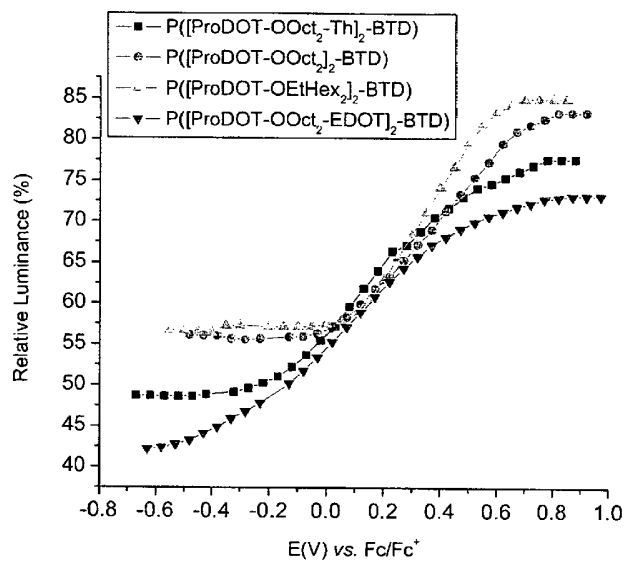


Figure 2

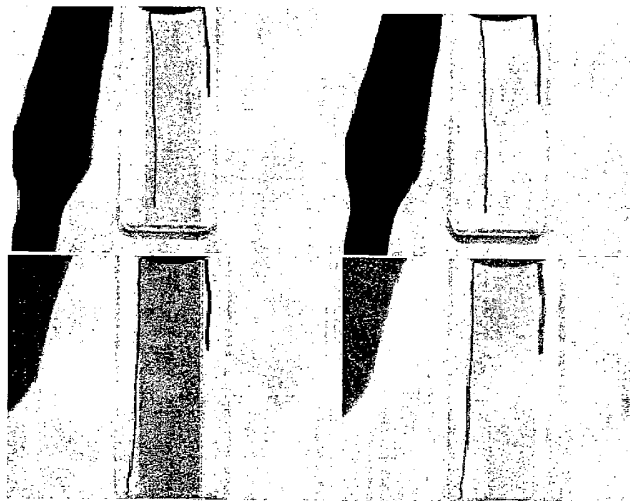


Figure 3A

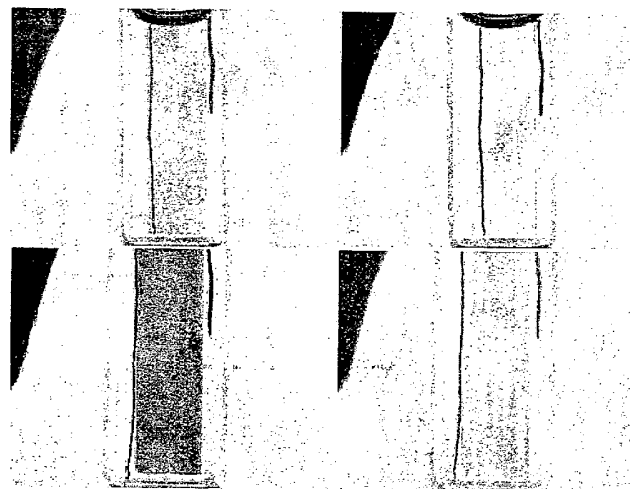


Figure 3B

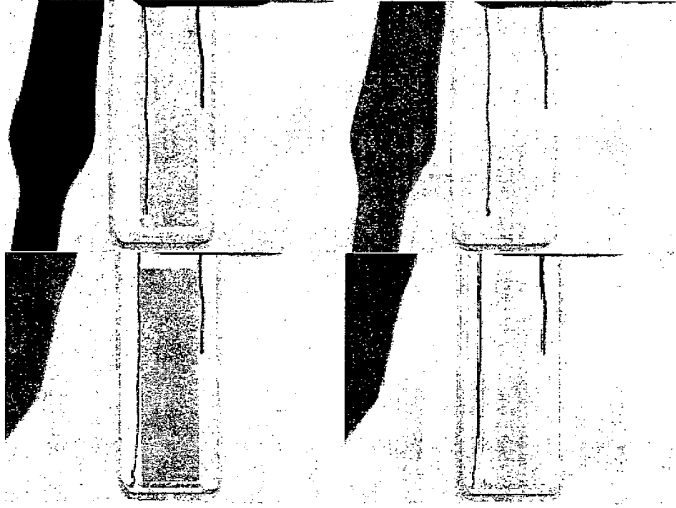


Figure 3C



Figure 3D