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High Contrast Electrochromism in Organic Materials

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14. ABSTRACT

Over the course of this program, we have developed both molecular and polymeric electrochromic materials with a special focus on understanding how the structure of conjugated systems can be altered to control radical cation and dication formation, and how this leads to optical transitions that manifest as a color change. Combining computational efforts, via collaboration with Prof. Aimée Tomlinson at the University of North Georgia, with the Reynolds group synthetic capabilities and characterization expertise has allowed us to deepen the level of understanding of cathodically coloring polymer electrochromes and break new ground in understanding the electronic and optical properties of novel anodically coloring electrochromes. Overall, we establish a more thorough fundamental understanding of redox behavior of conjugated systems, as we begin to see a growing interest in applying conjugated systems for mixed transport applications. In the area of polymer electrochromes, we highlight our development of a high-gap orange-to-clear polymer, demonstrating stability of thousands of switches, as well as the ability to be blended with other colors to attain black-to-clear electrochromic inks. With respect to fundamental work in the field of organic electronics, we deepened the understanding of how subtle structure property relationships – specifically side-chain tuning – impacts the performance of conjugated polymers in electrochemical and mixed-transport systems, demonstrating sub-second switching of electrochromic polymers in aqueous electrolyte for tens of thousands of switches. Additionally, we elucidated electrochemically charged states of conjugated polymers, proposing a model wherein formation of a charge-transfer state precedes formation of radical cations. Finally, we have developed a family of anodically coloring electrochromes, showing how straightforward synthetic handles can tune the optical absorbance of the oxidized state in predictable ways.

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"High Contrast Electrochromism in Organic Materials"

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Abstract:

Over the course of this program, we have developed both molecular and polymeric electrochromic materials with a special focus on understanding how the structure of conjugated systems can be altered to control radical cation and dication formation, and how this leads to optical transitions that manifest as a color change. Combining computational efforts, via collaboration with Prof. Aimée Tomlinson at the University of North Georgia, with the Reynolds group synthetic capabilities and characterization expertise has allowed us to deepen the level of understanding of cathodically coloring polymer electrochromes and break new ground in understanding the electronic and optical properties of novel anodically coloring electrochromes. Overall, we establish a more thorough fundamental understanding of redox behavior of conjugated systems, as we begin to see a growing interest in applying conjugated systems for mixed transport applications. In the area of polymer electrochromes, we highlight our development of a high-gap orange-to-clear polymer, demonstrating stability of thousands of switches, as well as the ability to be blended with other colors to attain black-to-clear electrochromic inks. With respect to fundamental work in the field of organic electronics, we deepened the understanding of how subtle structure property relationships – specifically side-chain tuning – impacts the performance of conjugated polymers in electrochemical and mixed-transport systems, demonstrating sub-second switching of electrochromic polymers in aqueous electrolyte for tens of thousands of switches. Additionally, we elucidated electrochemically charged states of conjugated polymers, proposing a model wherein formation of a charge-transfer state precedes formation of radical cations. Finally, we have developed a family of anodically coloring electrochromes, showing how straightforward synthetic handles can tune the optical absorbance of the oxidized state in predictable ways.

Introduction:

In the field of conjugated materials for electrochromic applications, much of the work that has been done has gone towards understanding how relatively large structural changes impact the color and switching properties. Through this approach, a large palette of colors has been developed that span the visible range. Of these materials, mid-gap (magenta/purple materials) and low bandgap (blues) systems have already attained a strong level of readiness, with contrasts > 70%, and cycling stability up to 100,000 cycles.

There are nevertheless a number of practical and fundamental areas where more work is required. First, high-gap systems with comparable contrast and cycling stability are at a lower level of readiness, with cycling stability typically on the order of 1,000 or fewer cycles. Second, the transmissive states of polymeric systems in particular are not truly colorless, often appearing somewhat gray, or slightly colored, due to light absorption by the charge carrier absorption band, centered in the near-IR, tailing strongly into the visible. Finally, a number of fundamental questions, especially related to the nature of the charged state, as well as how subtle structural modifications fine-tune electronic and optical properties, require deeper understanding.

Throughout our work in this program, we have tackled each of these problems through a collaboration with Aimée Tomlinson's computational group at the University of North Georgia. This partnership has allowed us to streamline and reduce the number of molecules we typically synthesize when trying to explore structure-property relationships. Additionally it has provided fundamental insight to understand the electronic transitions that give rise to the optical properties we observe.

In addition to continuing our polymeric work, we have developed a family of two-ring and three-ring molecular electrochromes based on dioxythiophenes. These materials have the advantage of possessing a truly colorless neutral state absorbing squarely in the UV. While small molecule electrochromic systems like viologens have previously been developed, these materials have not been able to cover as extensive a color palette. Additionally, there is not a deep fundamental understanding of the structure-property relationships governing the energy levels and optical properties of oxidized states in conjugated systems. In this program, we have worked to provide greater insight to begin putting together a set of design rules leading to structural handles for tuning the absorption bands and energy levels that will be applicable and advantageous for the entire field of organic electronics.

Experiment(s):

Synthesis and characterization: coupling reactions to prepare polymers and small molecules were primarily carried out using **direct arylation** (advantageous for its reaction scalability, being straightforward to set up, as well as its tolerance of a wide range of conditions), and **Stille coupling**. Molecules and polymers were characterized using **NMR**, **GPC**, and **elemental analysis**.

Electrochemical measurements were performed using a potentiostat to carry out cyclic voltammetry (**CV**), differential pulse voltammetry (**DPV**), **in situ conductance**, and electrochemical impedance spectroscopy (**EIS**).

Spectral measurements were performed using an Ocean Optics and Cary spectrophotometer to obtain optical **absorbance/transmittance** measurements as a function of applied potentials, and **switching speed/stability** studies observing the transmittance for a given wavelength as a function of time. Spectral measurements were often taken in conjunction with systems under potentiostatic control.

Thin films were coated using blade and spray coating and analyzed in cuvettes. Solution electrochemistry is measured using a custom optically transparent thin layer electrode (OTTLE) cuvette to minimize the path length for diffusing molecules.

Grazing incidence wide-angle x-ray scattering (**GIWAXS**) was carried out at SLAC and provided insight into the structural order in our polymers.

Raman and **transient absorbance spectroscopy** were carried out to investigate electrochemically induced charged states.

OECT device testing using a Keithley Sourcemeter was carried out in salt water to evaluate the mixed transport properties of glycolated conjugated polymers.

Time-dependent density functional theory (**TD-DFT**) was carried out by Aimée Tomlinson at the University of North Georgia to understand neutral and excited state transitions in our molecular electrochromes.

Results and Discussion:

This section will be divided into three main areas of focus: cathodically coloring polymer electrochromes, work on conjugated polymers that deepens the fields' fundamental understanding for use in mixed transport applications, and anodically coloring molecular electrochromes.

Cathodically Coloring Polymers

In this program, we developed two key areas necessary to bring cathodically coloring polymer technology closer to readiness. The first is deepening our structure/property understanding of high-gap polymers. In our prior work, we designed yellow polymers based on ProDOT-phenylene copolymers, taking advantage of the aromaticity of the phenylene ring to promote larger dihedral angles between the subunits which gives rise to higher bandgaps. These polymers, however, had high oxidation potentials, leading to poor switching stability (loss of color after <10 switches). Here, we changed our approach, utilizing all-thiophene copolymers and relying on steric interactions to induce torsional strain, while maintaining lower oxidation potentials compared to copolymers containing phenyl rings. Of these polymers, we focused on an orange-yellow dialkylthiophene-ProDOT copolymer that paired bulky groups on the thiophene with 1,3-methyl substitution on the ProDOT propylene bridge. To demonstrate this polymer's performance in an applied setting, devices were fabricated and cycled 10,000 times with loss of less than 5% contrast.[3] From this work, it was evident that small changes to the dioxythiophenes gave rise to notable differences in hue. Working with the Tomlinson group, we gained a clearer picture of how subtle changes to alkyl substitution and control of stereoisomer could predictably impact the neutral state color of our polymers, from which a palette of orange to red polymers were made.[10]

The second key area was in improving the state of the art of black-to-colorless switching conjugated polymer electrochromic systems that is of interest to so many technologies. Two areas targeted for improvement are enhancing the contrast, and having a more color neutral transition between the colored and colorless states. Much of our previous work on black-to-colorless switching has been directed to designing random copolymers with broad absorption. Using the high-gap orange-yellow dialkylthiophene-ProDOT developed above, along with four other polymer systems developed in our group – green (dual-band), cyan (dual-band), magenta (mid-gap), and yellow (high-gap) – we prepared various solution blends combining a high-gap, a mid-gap, and a dual-band polymer. This allows for more customizable tuning of blend ratios to afford specific hues of black. By selecting blend components with comparable oxidation potentials, this gives rise to components that oxidize/reduce at the same time, affording more color neutral transitions, achromatic neutral states ($C^*ab < 5$) and integrated optical contrast exceeding 40% across the entire visible spectrum (380–780 nm).[1]

Finally, we demonstrate how a range of hues from oranges through purple to blue can be accessed through using only three building blocks and controlling their feed ratios during polymerization. This approach aims to provide a practical approach to accessing a palette of colors, rather than needing to synthesize diverse building blocks for each desired hue. To further enable this approach, we demonstrate how TD-DFT can accurately predict the ratios needed for the polymerization to attain the desired color.[5]

Fundamental Studies and Aqueous Compatibility in Electrochromic Polymers

In the work described above we focused heavily on tackling problems necessary to bring electrochromic technology to a state of readiness. Nevertheless, there remain many fundamental questions that need addressing. Electrochromic properties that govern the switching speed are far less understood, for example. Through exploring a family of ProDOTs varying only in subtle differences on the side chains, we find that while the absorbance spectra are relatively the same among all samples tested, redox properties are more notably impacted. Of particular interest for electrochromic applications, the use of bulky side chains led to enhanced in-situ conductance, while enhancing polarity in the side chains led to more rapid ion transport, which in turn led to faster switching kinetics.[4] Building upon this, we further tuned the side chain to explore the aqueous electrochemistry of electrochromic polymers through using glycolated side chains of various lengths to better understand the mixed transport properties of conjugated polymers. In addition to extremely rapid switching (< 0.5s) we also observed excellent cycling stability, and OECT performance comparable to the state of the art. In the field of OECTs, this is also particularly impactful as much of the work has been focused on large backbone changes, while this represents the large impact small changes to the side chain length can have on performance.[7] Finally, we pursued a fundamental study of our glycolated polymers in aqueous, as well as organic aprotic, media to understand the mechanism of electrochemical doping. In contrast to well-established chemical doping mechanisms, electrochemical doping occurs in the presence of solvent and electrolyte, which offers a distinctly different environment. Through the use of resonance Raman and transient absorbance spectroscopy, we demonstrate the formation of a novel charge-transfer state occurring prior to, but near, the onset of oxidation, stabilized through polarization from the environment. These findings directly impact our understanding of polymer/electrolyte interactions, and emphasize the need to consider these interactions when looking to understanding the impact of subtly distinct side chains on various properties such as switching speeds and conductance.[9]

ACE Molecules

While we, and the field at large, have largely focused on developing an understanding of tuning electronic properties of conjugated systems in the neutral state, here we turn our focus to gain a deeper fundamental understanding of the structure-property relationships governing the oxidized states. To accomplish this, we have throughout this program worked closely with Aimée Tomlinson's computational group to rapidly screen structures, and to understand which energy levels give rise to the observed spectra. We began with a simple two-ring phenylene-EDOT system, tuning the functional groups attached to the phenylene ring to vary electron donating or withdrawing strengths. In the neutral states, all systems are colorless, absorbing in the UV with L*a*b* values of 100, 0, 0, while in their oxidized states the absorbance is comprised of two peaks that are red-shifted into the visible and near-IR. From computational and experimental results, we observe that changing the substitution pattern allows us to predictably control the absorbance in the oxidized state by modulating the position of the second lower-energy band, while minimally impacting the higher-energy band. Using this approach green, yellow and red anodically coloring electrochromes were developed and their switching observed in solution using a custom designed optically transparent thin layer electrochemical cuvette (OTTLE), where blends also give rise to a colorless-to-black solution.[6]

Building on this work, we expanded our study to include three-ring systems comprised of a phenylene center flanked by EDOTs, where the phenyl ring consisted of varying numbers and substitution patterns of methoxy groups. Through a combination of computational studies and crystal structures, these methoxy groups were observed to undergo sulfur-oxygen interactions that decreased the interatomic spacing and planarized the molecule. Conversely, destabilizing oxygen-oxygen repulsions gave rise to the opposite effect. These interactions in turn allowed for predictable control of the width of the high-energy peak in the oxidized state, which absorbs in the visible, providing us with another tool for tuning the anodic coloration of small molecule electrochromes. This gave rise to a family of colorless-to-blue electrochromes, where the vibrancy of the hue, dictated by the width of the peak, was varied.[11]

With the prior two studies, the electrochromic properties were evaluated with the molecules dissolved in solution. While this approach is adequate for understanding the agreement between computationally derived and experimentally observed spectra, for practical applications there are advantages to finding a strategy for immobilizing the dye on the electrode as a thin film. In solution, the oxidation state of the molecules can only be controlled at the surface of the electrode. When molecules diffuse back into the bulk of the solution, they are no longer under user control. This prevents electrochromes in the colored state from being rapidly driven back to the colorless state, for example, relying instead on the rate at which the electrochromes find their way back to the electrode. In contrast, molecules immobilized at the surface can be more rapidly controlled. Coated thin films tend to be impractical, as molecules are often soluble in the electrolyte in either their neutral or oxidized states. Instead, we synthetically attach functional groups that serve to covalently bind these molecules to the surface of oxide electrodes, an approach which has been well-developed in the field of DSSCs, and previously also used for anchoring viologen electrochromes onto TiO_2. Specifically, we functionalize our electrochromes with phosphonic acid groups which anchor onto ITO using a simple immersion in electrochrome solution for < 15 minutes. An EDOT-phenylene showed colorless-to-green switching within seconds, and little loss of contrast over 100 switches, demonstrating rapid user-control using this approach, as well as robust anchoring of the molecule over repeated redox cycling. In conjunction with functionalizing the electrochrome, it was also necessary to develop a way for sufficient quantities of chromophore to be anchored, as anchoring only attaches a monolayer of material which is insufficient for a vibrant hue to be observed. Adapting a procedure from the Meyer group at UNC, the surface of a smooth ITO/glass slide was coated with ITO nanoparticles to produce a mesoporous layer that enhanced the surface area by a factor of 30-50, with the precise amount controllable by tuning the coating parameters. This allowed sufficient electrochrome to be adsorbed to give rise to a notable colorless-to-green electrochromic switch.

2018-2021 AF Funded Peer Reviewed Publications and Patent Applications

1. Savagian, Lisa. R.; Österholm, Anna. M.; Shen, D. Eric; Christiansen, Dylan T.; Kuepfert, Michael; and Reynolds, John R.; Conjugated Polymer Blends for High Contrast Black-to-Transmissive Electrochromism. Adv. Opt. Mater. 2018, 1800594.

2. Christiansen, Dylan T.; Wheeler, David L.; Tomlinson, Aimée L.; and Reynolds, John R.; Electrochromism of Alkylene-linked Discrete Chromophore Polymers with Broad Radical Cation Light Absorption Polymer Chemistry; 2018, 9, 3055-3066.

3. Christiansen, Dylan T. and Reynolds, John R.; Fruitful Usage of a Dialkylthiophene for Redox Stable Switching in Wide-Gap Cathodically Coloring Electrochromic Polymers Macromolecules; 2018, 51, 22, 9250-9258.

4. Anna M. Österholm, James F. Ponder Jr., Michel De Keersmaecker, D. Eric Shen, John R. Reynolds, Disentangling Redox Properties and Capacitance in

Solution-Processed Conjugated Polymers, Chem. Mater. 2019, 31, 2971-2982.

5. Christiansen, Dylan T.; Ohtani, Shunsuke; Chujo, Yoshiki; Tomlinson, Aimée L.; and Reynolds, John R.; All Donor Electrochromic Polymers Tunable Across the Visible Spectrum: Calculations to Random Copolymerization, Chem. Mater., 2019, 31, 6841-6849.

6. Christiansen, Dylan T.; Tomlinson, Aimée L.; and Reynolds, John R.; New Design Paradigm for Color Control in Anodically Coloring Electrochromic Molecules, Journal of the American Chemical Society; 2019, 141, 9, 3859-3862.

7. Moser, M.; Savagian, L.R.; Savva, A.; Ponder, J.F.; Hidalgo, T.C.; Ohayon, D.; Hallini, R.; Reynolds, J.R.; Wadsworth, A.; Inal, S.; McCulloch, I. "Impact of Ethylene Glycol Side Chain Engineering on the Performance of Organic Electrochemical Transistors." Chem. Mater. 2020, 32, 6618−6628.

8. Nhon, L.; Wilkins, R.; Reynolds, J.R.; Tomlinson, A. “Guiding synthetic targets of anodically coloring electrochromes through density functional theory” J. Chem. Phys. 2021, 154, 054110.

9. Bargigia, I.; Savagian, L.R.; Österholm, A.M.; Reynolds, J.R.; Silva, C. “Charge-Transfer Intermediates in the Electrochemical Doping Mechanism of Conjugated Polymers” J. Am. Chem. Soc. 2021, 143, 294-308.

10. Collier, G.S.; Wilkins, R.; Tomlinson, A.L.; Reynolds, J.R. “Exploring Isomeric Effects on Optical and Electrochemical Properties of Red/Orange Electrochromic Polymers” Macromolecules 2021, 54, 1677-1692.

11. Nhon, L; Tomlinson, A. L.; Reynolds, J.R. Bis-3,4-ethylenedioxythiophene(BEDOT)-Aryl Discrete Chromophores for Anodically Coloring Electrochromic Applications. *In Preparation, Targeting Chemistry of Materials*.

Patent Applications and Patents

Reynolds, John R. and Christiansen, Dylan T.; US Patent Application US 2020/0393732 A1, December 17, 2020 PCT Patent Application No. PCT/US18/62043 filed November 20, 2018, “Anodically Coloring Electrochromic Molecules, Materials, and Devices, and Methods of Making and Use Thereof”

Conference Presentations by the PI John Reynolds

1. Gordon Research Conference, Electronic Processes in Organic Materials, Renaissance Tuscany Il Ciocco, Italy, July 2018.
   "Conjugated Polymer Design for Solid State and Redox Property Optimization"
2. International Meeting on Electrochromism (13), Chiba, Japan, August 2018.
   "Color Chemistry in Electrochromic Polymers"
3. Material Research Society, Boston, MA November 2018.
   "Polar and Reactive Side Chain Functionalization of Conjugated Polymers for Redox and Bio- Electronic Applications"
4. Material Research Society, Boston, MA November 2018.
   "Addressing the Problem of Black to Transmissive in Electrochromic Polymers for Switchable Windows"
5. American Chemical Society, 257th National Meeting & Exposition Orlando, FL March 2019.
   "Applied Materials and Interface Studies of Conjugated Electroactive Polymers"
6. F-π-14 International Symposium on Functional π-Electron Systems, Berlin, Germany, June 2019.
   "Fade to Black: Color Control in Electrochromic Polymers"
7. European Conference for Molecular Electronics (ECME), Linköping, Sweden, July 2019.
   "Conjugated Polymers in Redox Active Devices: Color, Charge, and Chemistry
8. American Chemical Society, 261st National Meeting & Exposition San Antonio, TX (Virtual) April 2021.
   "Conjugated Organic Molecules, Oligomers and Polymers"

Seminars presented by the PI John Reynolds

1. Ecole Normale Superieure de Saclay, Paris, France, May 2018
2. University of New Mexico, Albuquerque, NM, October 2018
3. Air Force Research Laboratory, Dayton, OH, November 2018
4. Mississippi State University, Starkville, MS, January 2019
5. University of Nevada at Reno, Reno, NV, April 2019

6. University of California, Santa Cruz, Sant Cruz, CA, April 2019
7. Purdue University, West Lafayette, IN, October 2019
8. University of Southern Mississippi, Covestro Lecture, Hattiesburg, MS, February 2020
9. Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta GA, February 2021

Presentations by colleagues of the PI on AF related research

1. 2018 Fall ACS National Meeting, Boston, MA, August 2018
   "Electrochromic Polymers Processed from Environmentally Benign Solvents" Graham S. Collier, Ian Pelse, Anna M. Österholm, and John R. Reynolds.
2. 13th International Meeting on Electrochromism, Chiba, Japan, August 2018
   "A New Method for Quantifying and Comparing Switching Time in Electrochromic Materials and Devices" with Anna M. Österholm, D. Eric Shen, Sofiane Hassab, Mathias Da Rocha, Giljoo Song, Yolanda Alesanco, Ana Vinuales, Aline Rougher, and Javier Padilla.
3. Materials Research Society, 2018 Fall Meeting & Exhibit, Symposium on Ions, Electrons, and Excitons in Organic Materials, Boston, MA, November 2018
   "Polar-Functionalized Dioxythiophene Copolymers as a Platform for Aqueous Compatible Bio-Electrochemical Devices," by L.R. Savagian, M. de Keersmaecker, J.F. Ponder, A.M. Österholm, and J.R. Reynolds.
4. 4th International Symposium on Functional π-Electron Systems, Berlin, Germany, 2-7 June 2019
   "Disentangling Redox Properties and Capacitance in Soluble Conjugated Polymers for Electrochemical and Bioelectronic Applications" by Österholm, A. M., Savagian, L. R., Ponder, J., Reynolds, J. R.
5. American Chemical Society, 261st National Meeting & Exposition San Antonio, TX (Virtual) April 2021
   "Exploring Isomeric Effects on Optical and Electrochemical Properties of Red/Orange Electrochromic Polymers" Graham S. Collier, Aimée L. Tomlinson, and John R. Reynolds.

Provide a list any interactions with industry or with Air Force Research Laboratory scientists or significant collaborations that resulted from this work.

1. Interactions continued with AFRL/RXAP (Nicholas Godman, Tod Grusenmeyer) - Interest exists in high contrast electrochromics for dimmable visors, laser protection, neutral color (black/gray/clear) electrochromism. Plans underway assuming re-continuation of the project.
2. Electrochromic Polymer and Device Technology, developed with AFOSR funded research used in 2020 with company Ynvisible for use in ITO-Free electrochromic devices. “Cost-Effective, Flexible, and Colorful Dynamic Displays: Removing Underlying Conducting Layers from Polymer-Based Electrochromic Devices”, *ACS Appl. Mater. Interfaces* **2021**, *13*, 16732−16743.
3. Electrochromic Polymers developed with AFOSR funded research being used in NASA funded switchable visor project by Giner Inc. (Avni Argun).
4. The color converter script (from UV-Vis to L*a*b* coordinates) was given to Joy Haley as per a request from Ruth Pachter.