DOI: 10.1002/anie.201205206

Color Modulation

# Reversible Full-Color Generation with Patterned Yellow Electrochromic Polymers**

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Structural colors in nature[1] are generally much brighter than chemical colors and commonly found in butterflies, beetles, and fish.[2] These colors change in some animals, such as the chameleon,[3] which camouflages itself by blending in with the surrounding colors. However in an artificial chameleon,[4] typically known as chromogenic system, it is difficult to achieve reversible color modulation as nature does, mainly because of the lack of materials or systems that show multicolor under different stimuli in a reproducible way. The structural colors change with respect to the angle of the structure to the eye, or with respect to the depth of the pattern,[5] however, keeping these parameters intact makes reversible color modulation is challenge.

In passive gratings,[6] diffractive colors are produced, but color switching is not possible since the refractive index of the grating material is fixed. To generate multicolor and to switch from one color to another, at least three or more gratings with different grating parameters (period and thickness) are required, which are then superimposed to produce a full color scheme. For example, Knop reported multiple-phase gratings using polyvinylchloride with different thickness over 400 nm to generate visible colors.[7] In a recent report, microchannels of poly(dimethylsiloxane) (PDMS) were used as a tunable visual color filter based on microfluidic transmission and a shift from red to blue color was observed.[8] The grating materials with different refractive index were allowed to flow through the microchannels to obtain a different colors. Ge et al.[9] employed colloidal photonic crystals having reversible tunability in response to external magnetic fields. Electroactive thin films patterned by photolithographic or imprint method have been explored for monochromatic diffraction intensity modulation[10] and in a recent report, electroactive subwavelength gratings are used for color and intensity modulation in reflective mode[11] however, there are hardly any reports on the use of patterned conjugated polymers as diffraction gratings for color modulation, despite wide range of conjugated polymers.[12] Thus we considered it challenging to explore electrochemical (EC) gratings as visual color filters and artificial chameleons. Electrochemically active polymers have a great advantage over other materials because of their ability to change redox state under external bias,[10–13] which results in a change in the refractive index.

Herein, we report a new method to obtain an artificial chameleon effect on reversibly electroactive polymer gratings without changing gratings parameters, employing multiple gratings, or changing the surrounding medium. Considering the light dispersion principles,[7] we hypothesized that a color generated from the dispersion of light into a polymer grating can be electrochemically modulated by changing the redox state of the polymer in the grating. Thus, the aim of this study is to demonstrate reversible color modulation by electrochemical reactions using a patterned film of propylenedioxythiophene phenylene copolymer P(ProDOT-Ph). This polymer is cathodically coloring,[14] changes color from yellow to pale blue depending upon applied voltage. We chose the yellow electrochromic polymer because it has a faint color and thus the optical absorption factor change, on applying a voltage, is mainly a result of the change in the refractive index because the extinction coefficient change in the imaginary part is weak.[13d] Moreover it shows a greater change in refractive index between the neutral and oxidized state in comparison to P3HT.[11] The change in refractive index was found to be almost two times more than that of P3HT in the small voltage range of −2 to 2 V.

The yellow electrochromic polymer P(ProDOT-Ph) was synthesized by a Suzuki coupling reaction following reported procedures (see Supporting Information, Figure S1–S4).[14,15] A solution with 0.4 wt% of the polymer P(ProDOT-Ph) in chloroform was used for the preparation of the thin film and patterning. The line grating patterns of the polymer were fabricated with the micromolding in capillaries (MIMIC) method.[16] The overall scheme for simple and large area (ca. 1.5 cm$^2$) patterning of P(ProDOT-Ph) using the elastomeric PDMS stamp is shown in Figure 1a. An elastomeric PDMS stamp with a line grating with a 10 μm period, 5.6 μm relief feature, and 1 μm pattern depth used was confirmed by scanning electron microscope (SEM) as shown in Figure 1b. The SEM image in Figure 1c shows the polymer gratings in large area created by MIMIC with an applied pressure of 0.0015 MPa. The patterns were of uniform thickness and were

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[**] This study was financially supported by a National Research Foundation (NRF) grant funded by the Korean government (MEST) through the Active Polymer Center for Pattern Integration (R11-2007-050-00000-0), the Pioneer Research Center Program (2011-0001672), and the Converging Research Center Program through the Ministry of Education, Science and Technology (2010K001430). This work was supported (in part) by the Yonsei University Research Fund of 2010.

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/anie.201205206.

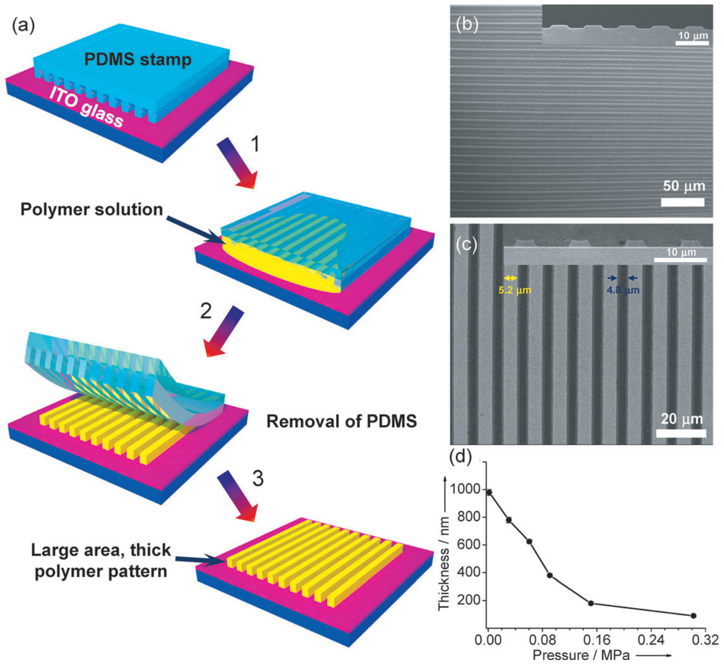

***Figure 1.*** a) The MIMIC method for the preparation of P(ProDOT-Ph) patterns: 1) Patterned PDMS stamp placed firmly on the ITO glass. Polymer solution was cast on one end of the PDMS stamp allowing the solution to fill the microchannels; 2) Solvent evaporation at room temperature; and 3) Detachment of PDMS stamp. SEM images of b) the patterned elastomeric PDMS stamp and c) the patterned P(ProDOT-Ph), inset: its cross-sectional profile. d) A plot of the applied pressure on the PDMS stamp against the thickness of the polymer pattern obtained by MIMIC method.

parallel to each other. The period of the polymer grating was 10 μm with a relief feature of 4.2 μm separated by a distance of 5.8 μm. The thickness of the pattern (ca. 980 nm) was found to reach near the depth of the stamp itself. This patterning process was modified by application of different pressures on the elastomeric PDMS stamp during patterning. The application of pressures ranging from 0.0015 to 0.03 MPa resulted in a modification of the thickness profile on the patterned polymer. Figure 1 d shows the effect of pressure on the thickness of the pattern. At higher applied pressure, the elastomeric stamp was compressed more, resulting in a decrease in the thickness of the channels in the stamp itself. When approximately 0.01 MPa of pressure was applied, the feature size decreased to half of the original thickness (Supporting Information, Figure S5). This method was preferred as it resulted in isolated thick strips of the polymer on the substrate, whereas surface patterning or imprinting methods resulted in a continuous residual polymer layer on the substrate between the gratings.

An electrochemical (EC) device was fabricated with the polymer film on indium-tin oxide (ITO) glass as a working electrode and bare ITO glass as a counter electrode. A liquid electrolyte consisting of a $0.2\,\text{M}$ solution of lithium bistrifluoromethanesulfonimide/propylene carbonate (LiBTI/PC) was filled between the two electrodes. Figure 2 shows the effect of the external potential on the optical properties of the EC cell. The cell with the unpatterned polymer film was yellow colored at −2 V at neutral state,[11,14] with an absorption at 440 nm with a low transmittance of 14 % as shown in Figure 2 a. When the applied voltage was 1.5 V, the absorbance at 440 nm dropped by 25 % and the absorption over the rest of the visible spectral range was slightly increased. As the voltage was further increased to 2 V (oxidized state), a small but broad absorption band appeared at around 620 nm along with a weak band at 440 nm and transmittance of 50 %. As shown in the inset of Figure 2 a and Figure S6 in the Supporting Information, optical images of the EC cell were vivid yellow at −2 V and greenish blue at 2 V. Such electrochromism originates from the reversible redox reactions described in Figure 2 c, in good agreement with previous report.[11,14]

For the EC cell with a patterned polymer film, there was no distinct absorption peak at around 440 nm (Figure 2 b) in comparison to the unpatterned film. Instead, the absorption band was observed at around 525 nm with transmittance of 38 %. Upon application of 2 V, the color of the cell was very weak and the transmittance increased to 60 %. As shown in the inset of Figure 2 b and Figure S6, optical images of the EC cell were pale yellow at −2 V and transparent greenish blue at 2 V. It would be expected that the absorbance of the patterned film should be higher than that of the unpatterned films owing to its greater thickness despite the fill factor being 0.42, but the absorption spectra

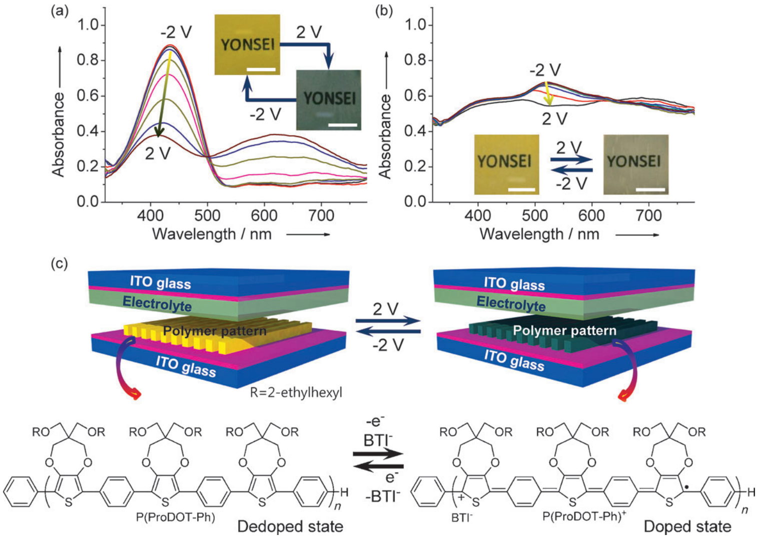

***Figure 2.*** Absorption spectra recorded at different voltages from −2, 0, 1, and 2 V (from 1 V, with 0.2 V increase) for a) a 180 nm thick unpatterned P(ProDOT-Ph) film and b) a 770 nm thick patterned P(ProDOT-Ph) film. Inset showing the optical images taken at −2 V (neutral) and 2 V (oxidized). Scale bar, 0.5 cm. c) The electrochromism arises from the reversible redox reactions in the EC cell (BTI = bis-trifluoromethanesulfonimide).

revealed that the patterned film is more transparent and uniform over the visible range as a result of diffraction through the patterned polymer.

When conducting polymers undergo reversible redox reactions, this process is usually accompanied by a change in the refractive index of the polymer which is a result of doping and de-doping of counterions from the electrolyte medium.[12b] Another important phenomenon that is also associated with the electrochemical reaction in these polymers is the volume change. The volume change is as high as 30–40 % for polypyrrorle derivatives[13b] whereas for poly(3,4-ethylenedioxythiophene) (PEDOT) derivatives it is less than 1 %,[13f] and hence in the present study the volume change in the polymeric matrix can be ignored. We opted to measure the change in refractive index by measuring the change in diffraction efficiency ($DE$). The $DE$ of a grating is determined from the ratio of the total diffracted intensity ($I_{\mathrm{diff}}$) and the incident light intensity ($I_0$) as $DE = I_{\mathrm{diff}}/I_0$ where $I_{\mathrm{diff}}$ is the sum of the intensity for all of the diffracted light. For a square-wave phase grating, the $DE$ of the zeroth order ($DE_{\mathrm{m=0}}$) ($m$ refers to the diffraction order) and first order ($DE_{\mathrm{m=1}}$) can be calculated based on the Equations (1)–(3)

$$DE_{m=0} = \cos^2(\xi/2) \tag{1}$$

$$DE_{m=1} = \{2/\pi \sin(\xi/2)\}^2 \tag{2}$$

$$\xi = 2\pi t\,(n_0 - n_1)/\lambda \tag{3}$$

where $\xi$ is the phase difference between the grating material and the surrounding medium, $t$ is the thickness of the grating, and $n_0$ and $n_1$ are the refractive index of the grating material and the surrounding medium, respectively. The line grating creates a spatially periodic modulation of the refractive index, and $\xi$ depends on the peak-to-peak amplitude path of the light. From Equations (2) and (3), by knowing $DE_{m=1}$ for a given $\lambda$, the change in the refractive index, $\Delta n = |n_0 - n_1|$, can be calculated. In the present study, the refractive index of the polymer can be reversibly changed by electrochemical doping and de-doping process. For a given thickness, the color of the polymer grating can be modulated conveniently in an EC cell, without changing the thickness of the polymer grating. When $\Delta n$ changes reversibly, it is possible to tune the color of the pattern reversibly and generate new color in the intermediate value of $\Delta n$.

The schematic of the experimental set up for $DE$ measurement is shown in Figure S7a in the Supporting Information. For calculation of the refractive index for the polymer in the neutral and oxidized states, the first order $DE$ was measured. The diffraction pattern at −2 and 2 V of the EC cell with a 770 nm thick polymer pattern is shown in Figures S7b,c. The central zeroth-order diffracted light and the adjacent first-order and second-order diffraction at −2 V were stronger than at 2 V. The first-order spots were equidistant from the zeroth-order spot implying the diffraction pattern is completely symmetric. The intensity of the two spots arising from the first-order diffraction was measured and they were found to be of similar intensity (with deviation less than 10 μW). $DE_{m=1}$ was determined as 0.35 and 0.11 for the polymer pattern at −2 and 2 V, respectively, using a 635 nm laser. Such a large variation in $DE$ value is ascribed to the large grating thickness (770 nm) and is important for color modulation in a cell. From the $DE_{m=1}$, the refractive index difference between the polymer pattern and electrolyte ($\Delta n$) was estimated 0.313 and 0.144 at −2 V to 2 V, respectively, implying the maximum $\Delta n$ of 0.169 within this potential ranges.

Results on color generation and modulation through the polymer grating are shown in Figure 3. A white light emitting diode (LED) light was used to illuminate the EC cell and the zeroth-order diffracted light was collected in transmission mode (see Figure 3 a) at applied voltage varying from −2 to 2 V. The spectrum of the white light source employed is provided in Figure S8 (see Supporting Information). The colors obtained for a 770 nm thick grating device at fixed angle of incidence and a 180 nm thick unpatterned film device at different voltages are shown in Figure 3 b and Figure S9. The color obtained for the grating device (Figure 3 b(1) and S9) was bright green at −2 V and it remained green until 0 V and upon application of 1 V, it turned to blue. In the intermediate doped states, different color mixing occurred such as blue color at 1.5 V and finally purple upon application of 2 V. In the case of the unpatterned film (Figure 3 b(2)), the transmitted light appeared as orange brown (−2 V) and bluish purple (2 V), which is different from the structural colors observed from the patterned film. This experiment distinguishes the color change by diffraction from electro-

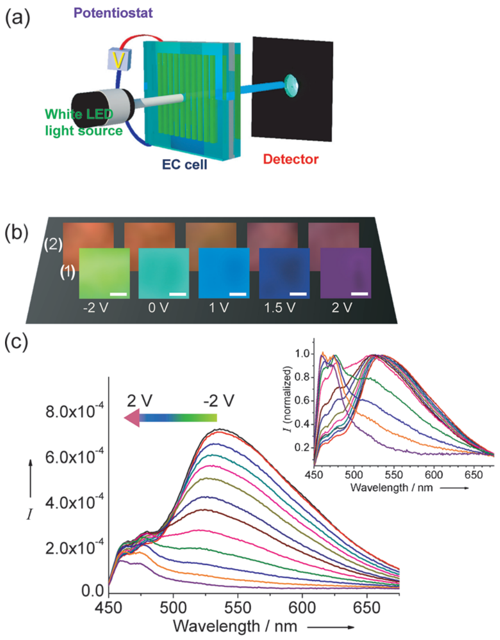

**Figure 3.** a) The experimental set up for the color generation in transmission mode. A white LED light illuminated the EC cell to generate the various colors under controlled applied voltage. b) Optical images of colors obtained at different voltages for 1) a 770 nm thick polymer grating and 2) a 180 nm thick unpatterned film under a white LED light source. Scale bar, 0.3 cm. c) Plot of diffracted light intensity ($I$) for a 770 nm thick polymer grating, at different applied voltages; −2, 0, 1, and 2 V (with 0.1 V increase between 1 V and 2 V) under a white LED light source. Inset: Plot for the normalized diffracted light intensity ($I$) at different voltages.

chromism. Different colors are generated on application of external potential on patterned polymer which is otherwise not possible with mere thin polymer film. Figure 3c shows the spectral change of the EC cell with a 770 nm thick polymer pattern by the voltage variation from −2 to 2 V at fixed angle (55°). Upon application of a voltage of −2 V, the $\lambda_{max}$ of the diffracted light of the EC cell was 536 nm and it changed to 460 nm at 2 V. The normalized diffracted light intensity obtained for −2 to 2 V are shown in inset Figure 3c. Indeed vivid colors were generated from the EC grating device when transmitted from a white LED light source.

With the change in the redox (doping) state of the polymer by the application of external voltage, $\Delta n$ changes causing $n_0$ to change. Color modulation with grating devices of different thickness is demonstrated in Figure 4. A plot of change in $\lambda_{max}$ value and $\Delta n$ on application of external potential for 770 nm thick grating device is shown in Figure 4a. The $\lambda_{max}$ and $\Delta n$ values did not change significantly between −2 and 0 V as the polymer was in neutral state, but they changed drastically as the voltage was varied from 1 to 2 V, since the polymer was electrochemically doped and this resulted in the change of refractive index of the polymer. At voltages from −2 to 2 V, the $\lambda_{max}$ value decreased from 536 and 460 nm. The $\Delta n$ change is correlated well with the $\lambda_{max}$ values at different voltages and especially the maximum change was observed between 1 and 2 V. Thus, the variation in $\lambda_{max}$ values is primarily due to $\Delta n$ change, which being a complex function shows little variation in the trend in $\Delta n$ change in comparison to $\lambda_{max}$ values (Figure 4a, inset). This emphasizes that $\Delta n$ change can be achieved by application of external potential and this can be utilized efficiently for color modulation in diffraction gratings without changing any grating parameters.

According to Equation (3), the color modulation should be more enhanced for thicker gratings. To elucidate this fact, the gratings devices with different thickness namely 100, 210, 430, 630, 770, and 980 nm were fabricated with a constant period of 10 μm. The change in $DE$ between the redox states was higher for thicker gratings, as indicated from the plot for the $DE$ change for the first-order diffraction, as a function of thickness at −2 and 2 V (Figure S10, see Supporting Information). The color ranges covered by the EC devices of different thickness are compared by colorimetric $a^*b^*$ (CIE 1976 $L^*a^*b^*$ Color Space)[17] color coordinates at different voltages as seen in Figure 4b. In this plot, $x$-axis represents the change from red to green moving from a positive to a negative value of $a^*$ and $y$-axis represents the color change from yellow to blue color moving from a positive to a negative value of $b^*$. It becomes very clear that unlike the unpatterned film (Figure 4b, black diamond), which only covered the yellow range, the patterned polymer (770 nm thick, green circle) had a wide range of color generation covering yellowish green to blue and to purple. The EC device with a 980 nm thick grating showed the widest range of colors than the others. Interestingly, all the patterns gave reddish magenta color, which clearly distinguished from the EC color of the unpatterned film.

Figure 4c illustrates the change in $\lambda_{max}$ value on application of external voltage between −2 and 2 V for devices with different thickness. As expected, there was no significant change in $\lambda_{max}$ value in the voltage range of −2 to 0 V. The change in $\lambda_{max}$ value on application of positive bias was maximum for 980 nm thick device, change in $\lambda_{max}$ value of 77 nm. Thus, $\lambda_{max}$ change was correlated to the applied voltage between −2 and 2 V for the EC device with different grating thickness. As the thickness of the grating decreased, the color range covered by the grating at the different voltage also decreased, as expected from Equation (3). Simulated spectra for $DE_{m=0}$ were plotted for the visible spectra of the EC device at −2 V with different grating thicknesses using Equation (1) knowing the refractive indices of the polymer and electrolyte (Figure S10a). The experimental results were quite comparable to that of the theoretical plot shown as a dotted line in Figure S10b, supporting the validity of Equation (3).

The experimental intensities were higher than the calculated values in the lower wavelength range (Figure S10b). This is probably due to the absorption peak in the yellow color range. The mechanism of color modulation in

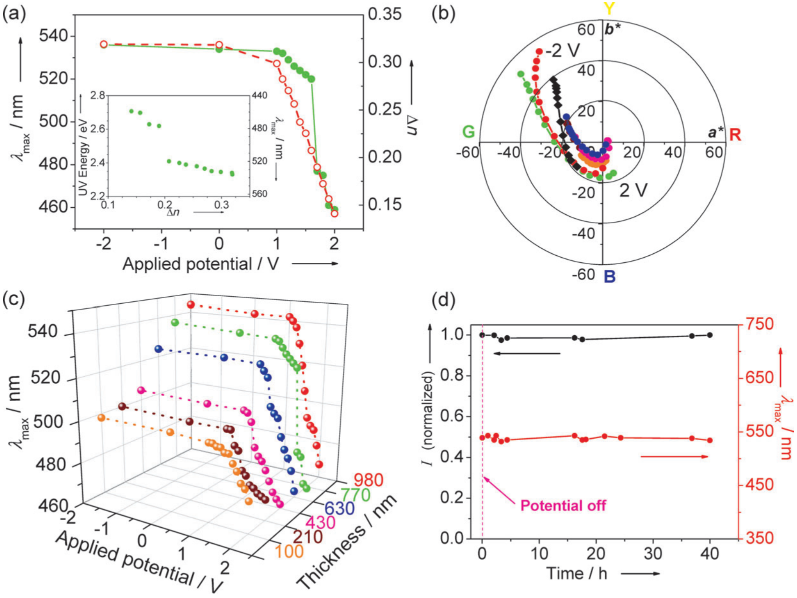

**Figure 4.** Comparison of the color change for the patterned and unpatterned device at different applied voltages. a) A plot of variation in $\lambda_{max}$ (green) and $\Delta n$ (red) for a 770 nm thick grating device as a function of applied voltage. Inset: Plot of $\lambda_{max}$ (in energy unit eV and wavelength unit nm, as a function of $\Delta n$). b) Colorimetric $a^*b^*$ color coordinates plot corresponding for the grating device of different thickness (orange 100 nm, brown 210 nm, magenta 430 nm, blue 630 nm, green 770 nm, and red 980 nm) compared to the unpatterned film with a thickness of 180 nm (black diamond) for different applied voltages. c) A plot of $\lambda_{max}$ for the grating devices of different thickness as a function of applied voltage. d) A plot of change in diffracted intensity ($I$) and $\lambda_{max}$ value for a 770 nm thick patterned EC device as a function of time illustrating the memory effect. The device was held at −2 V before the power supply was turned off.

this EC device is based on reversible electrochemical doping reactions of the polymer, which offers stability at different voltages. The oxidized or neutral states are stable even after removal of electric power supply. As shown in Figure 4 d, the diffracted color and intensity of the EC grating device at $-2$ V was maintained even after the electric power supply was turned off. In the power-off state, the decrease in diffracted intensity was less than 10 % even after 40 h. This memory effect is important for display devices which will allow less consumption of power. Thus, the present study clearly elucidates that the EC devices using patterned electroactive polymers film can induce a reversible color change along with a refractive-index change through electrochemical doping/dedoping. The application of external potential gives control over the modulation of desired color. This also demonstrates a simple method for vivid color generation in the EC device which is otherwise not possible by mere use of electroactive thin film without any patterns.

In summary, a yellow electrochromic polymer was successfully patterned with the fast and simple MIMIC process to fabricate large-area thick-line gratings. Under the illumination of white LED light, the diffraction gratings exhibited different colors upon application of an external potential. For the 770 nm thick grating, the color varied from green to magenta with an intermediate blue color upon application of $-2$ to 2 V thus covering the visible spectral range (RGB) and which was not achieved with an unpatterned film. Furthermore, this work demonstrates how the change in the refractive index of an electroactive polymer upon application of an external potential can be exploited for electroactive functional devices. With complete reversibility and color modulation on applying a voltage, the grating device holds the key to the artificial chameleon effect.

Received: July 3, 2012
Revised: August 21, 2012

**Keywords:** color modulation · diffraction · doping · electrochemistry · electrochromic polymer

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