

White Electrofluorescence Switching from Electrochemically Convertible Yellow Fluorescent Dyad

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A fluorescent naphthalimide-tetrazine dyad (NITZ) was examined for electrofluorochromism. The reversible electrochemistry of the tetrazine was accompanied by the fluorescence change through a quasi-complete energy transfer in an electrochemical cell prepared by the mixture of polymer electrolyte and naphthalimide-tetrazine dyad. Owing to the energy transfer within the dyad (naphthalimide and tetrazine), the fluorescence efficiency of NITZ was much enhanced and the effective fluorophore concentration in this system was much less than other tetrazine based electrofluorochromic device (EFD). Thus the yellow fluorescence of NITZ was switched on and off remarkably even with small quantity of NITZ (1 wt.%) in an EFD upon application of step potentials for different redox state. Furthermore, multi-color fluorescence switching was achieved by blending a naphthalimide to the electrofluorochromic layer, to show white-blue-dark state of fluorescence. Since the tetrazine and naphthalimide units have their emission quenched at different potentials, the emission color could be tuned by quenching emission at selected wavelengths, reversibly, under low working potentials.

cases where the same moiety acts both as the light emitter and the redox switch are much rarer,^[11–13] especially in the field of organic dyes. A few examples have been described, by us^[11–19] and others^[6–8,20,21] one of them implying a multistep process with a molecular internal rearrangement.^[7,8] Tetrazine derivatives that are currently developed in our group constitute a family of very promising candidates for such applications.^[22–27]

We have published recently reports describing all solid-state electrofluorochromic devices, demonstrating the ability to reversibly switch the fluorescence properties according to the voltage applied to two electrode plates between which the redox active fluorophore layer was incorporated as the cathodic material, which lead to a two-state switchable device.^[12,13] A further challenge now consists of obtaining more than two states, and especially to be

able to obtain with the same device, aside from one “off” state, several “on” states displaying different colors. This should be in principle possible by using dyad-type chromophores, and or mixtures of fluorophores.^[28,29] However, one has to take care that there is no mutual fluorescence quenching by one fluorophore on the other one; in addition, both should be electro-switchable in the same potential region. That is, both should be oxidizable or reducible, so that fluorescence quenching through charge transfer be excluded as much as possible. Therefore NITZ is a very promising candidate to examine electrochemical fluorescence switching, in which the high fluorescence of the neutral state of the chromophore could be reversibly switched on and off by converting the molecules successively to their reduced form (non fluorescent) and back to the neutral (fluorescent) state.^[11–13] Here being a dyad, there are three different states; neutral NITZ, tetrazine (TZ) reduced state, and both TZ and naphthalimide (NI) reduced states. Thus it is interesting to explore how the fluorescence of the second fluorophore (NI) can be visualized into a device also containing NITZ as a second fluorophore, to achieve multi-color fluorescence at variable redox states controlled by the external potential. Since the dyad is very bright due to energy transfer between the partners, it shines more than the parent imide. Therefore when the tetrazine in the dyad is reduced, this does not affect the fluorescence of the imide which is present in the blend with a higher concentration. When the concentrations are carefully selected, a three-color EFD, respectively white, blue and non-emissive, can be obtained. In this paper we therefore describe the first

1. Introduction

Reversible switching of optical status by electrochemical or photochemical conversion of molecular energy state is an attractive field of research due to the high interest in controlling reversible optical memories,^[1,2] displays,^[3,4] or signalling recognition events.^[5,6] To achieve these research demands, electrically driven reversible luminescence switching can be a promising candidate. A few examples already exist in the literature in which the fluorophore emission can be switched by changing the redox state of an active site connected to it,^[6–10] but the

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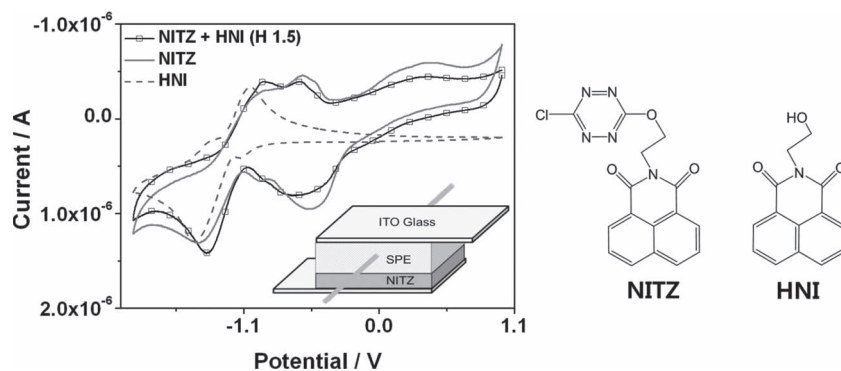


Figure 1. Cyclic voltammogram of NITZ (solid line), NITZ blend with HNI (solid, open square), and HNI (dashed line) in solid polymer electrolyte, recorded with a scan rate of 20 mV/s in 3-electrodes switching cell with an Ag wire reference. Inset shows the structure of the three-electrode device with a thin NITZ layer, a silver wire, and a SPE layer. The chemical structures of NITZ and HNI are shown.

example of a three-state device using a bright naphthalimide-tetrazine dyad,^[27] in blend with the parent imide.

2. Results and Discussion

2.1. The Redox Properties and Electrofluorochromic Switching of NITZ in a Solid State Cell

The cell with NITZ is a sandwiched device made of two layers packed between two transparent ITO electrodes (Figure 1). The first layer, a layer of a viscous polymer electrolyte solution was coated on an ITO plate by spin coating and then photo-cured so that it became a stiff solid polymer electrolyte (SPE) film. Then a layer of the polymer electrolyte solution containing 1 wt% of NITZ was coated on the other ITO plate. The two plates were then contacted, sealed with a reference electrode in a 3-electrodes system. The redox potentials of NITZ in solid polymer electrolyte (SPE) media in 3-electrodes system were observed at -0.67 V and -1.58 V (red line in Figure 1), for the reduction wave of the tetrazine (TZ) and the naphthalimide (NI) unit, respectively, which were well matched to those in liquid electrolyte (Figure S1).^[27] These two reduction waves were reversible and reproduced by precise measurement.

The absorption and fluorescence spectra of NITZ in SPE, without application of potential, matched very well those in liquid system. Two absorption bands were observed at 334 (strong) and 518 nm (weak) for the naphthalimide and the tetrazine unit, respectively, which resulted in two fluorescence bands at 385 (weak) and 558 nm (strong), respectively. Although the absorption in the visible region (max. 518 nm) of the NITZ was weak, due to its $n-\pi^*$ transition, it displayed a strong fluorescence, while the excitation at 355 nm, where the

naphthalimide absorbs almost exclusively, the corresponding fluorescence (maximized at 385 nm) was almost disappeared, due to the occurrence of energy transfer between the two chromophores (NI to TZ units). In the previous report, the efficiency of the energy transfer was calculated to be 95%,^[27] which could be similar in the SPE media as the optical properties of NITZ in SPE were well matched to that in dichloromethane.

The electrofluorochromic switching of NITZ was examined by monitoring the photoluminescent properties at different applied potentials (Figure 2a) with a three-electrode switching device using a solid polymer electrolyte layer (SPE). The cell showed vivid yellow fluorescence without application of potential or when the applied potential to the cell was positive (Figure 2a, inset). It was

noteworthy that the minimum content of NITZ to observe the fluorescence was much less than that of typical alkoxy tetrazine (e. g. ~ 12 times less than that for methoxy tetrazine) owing to the high fluorescence quantum yield of the dyad (NITZ).^[12] Upon application of a negative potential below zero, the fluorescence was quenched and the cell was significantly extinguished to dark when the applied potential was beyond -1.0 V and then almost completely extinguished after -1.4 V (Figure 2a, inset). The fluorescence intensity change occurred without a shift of the spectral band with the potential change (Figure 2a), indicating that the fluorescence quenching originated from the electrochemical reduction of the neutral fluorophore (TZ) to its anion-radical form, without the production of side products.

Interestingly, when the applied potential was more negative than the reduction potential of TZ but less than that of NI ($-0.6 \sim -1.2$ V), where the TZ unit should be reduced but NI be in neutral state, the fluorescence intensity of TZ (max. 558 nm) decreased. Moreover, the blue fluorescence from NI units was not observed in any stage of reduction even after TZ units were completely reduced (> -1.0 V). After -1.0 V, although very weak, the fluorescence from the cell was still observed as dark

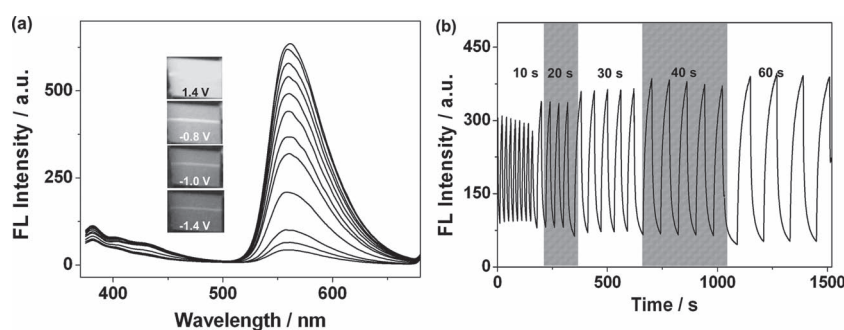


Figure 2. (a) Fluorescence changes of a three-electrode switching cell containing NITZ at different applied potentials from +1.4 V (top) to -1.4 V (bottom) with 0.2 V decrease at each step. Each spectrum was obtained after applying the target potential for 60 s to obtain the fluorescence spectra at saturated state (excitation at 355 nm). Inset: Fluorescence switching image of the NITZ at 1.4 V, -0.8 V, -1.0 V and -1.4 V. (b) Fluorescence switching responses of the cell monitored at 558 nm under applied step potential of 1.4 V to -1.4 V with a step duration time for 10 s, 20 s, 30 s, 40 s, and 60 s at each potential.

yellow, indicating the occurrence of energy transfer from NI to TZ, similarly observed at the neutral state. The yellow emission from the energy transferred state of NITZ was almost completely extinguished only after -1.4 V, beyond which the cell was dark as shown in the photographs of Figure 2a, inset.

The fluorescence response to the applied potential was reversible as examined from the recovery of the intensity with the application of the reverse potential toward positive one to lead to neutralization of NITZ. Figure 2b shows the reversible electro-fluorescence switching upon repetitive cycling between $+1.4$ V and -1.4 V, beyond which the reversibility between the dark to vivid yellow fluorescent state (neutral state) was poor. Thus in this experiment, the potential was quickly scanned without waiting for the full reduction of NITZ which explains why the variation of intensity is smaller than expected from the spectral change in Figure 2a. Indeed it became larger when the duration time at each step potential was longer as shown in Figure 2b.

The fluorescence switching in the study originates from the reversible redox properties of NITZ sandwiched between a photocured SPE and an ITO electrode. As there is an efficient energy transfer between the TZ and NI, the fluorescence from NI unit was not observed in the intermediate potential range ($-0.6 \sim -1.0$ V). Owing to the energy transfer, the fluorescence efficiency of the dyad (NITZ) was much enhanced and it shines more than the parent imide. The quantum yield of NITZ and HNI was reported as 0.32 and 0.06, respectively.^[27] Thus the effective fluorophore concentration in this system is much less than other tetrazine based EFD. To visualize NI fluorescence enough to achieve multi-color fluorescence in an EFD, therefore, we added (2-hydroxyethyl)-N-naphthalimide (HNI) because the reduced NITZ would not affect the fluorescence of the rest of the naphthalimide which is in the blend with a high concentration.

2.2. The Electrofluorochromic Switching of NITZ Blend with HNI (Three States)

The redox peak of (2-hydroxyethyl)-N-naphthalimide (HNI) in SPE was observed at -1.6 V, which was overlapped with the reduction peak of NI unit in NITZ (Figure 1). The blue fluorescence of HNI at neutral state was quenched as HNI was reduced and then reversibly turned on blue fluorescence when HNI was electrochemically neutralized. Since HNI also shows quasi-reversible fluorescence switching response upon the external potential, a tetrazine blend containing NITZ and HNI was examined for a multi-color fluorescence switch through careful control of the redox state of the fluorophore blend. Three electro fluorescent devices (EFDs) were prepared using NITZ-HNI blends having different HNI content and compared with the pure NITZ system (H0). The redox properties of the blend occurred as expected from

the individual processes of the components, with no apparent interference of one compound on the behavior of the other one, as it could be expected. Interestingly, the device with NITZ-HNI blends showed the emission from HNI at neutral state.

The optical properties of these blend was also the same as the individual components. No side product formation was noticed (Figure 3a), indicating that the extra HNI is untouched by the energy transfer between TZ and NI. On the other hands, when the content of HNI was more than twice of NITZ, the fluorescence of the TZ part was reduced (Figure 3a), partly due to the dilution effect by the additional HNI. Thus we examined electrofluorochromic colors of the cell with the NITZ: HNI ratio of 1: 0.5 \sim 4 [1 : 0.5 (H0.5), 1 : 1 (H1.0), 1 : 1.5 (H1.5)].

As shown in Figure 3b, both fluorescence bands for TZ and NI unit, maximized at 558 and 385 nm, respectively, were decreased according to applied potential toward negative. Interestingly, the TZ emission was decreased first up to -1.0 V and then the NI emission was decreased when the applied potential were below -1.2 V. This result is well understood from the redox reaction of TZ and NI, of which reduction are peaked at -0.8 V and -1.6 V, respectively. Thus the TZ unit is reduced first to extinguish the emission from TZ (max. 558nm) until the applied potential reaches to -1.2 V, and then NI unit reduced afterwards, to quench the emission from additional HNI. These results were more quantitatively shown in Figure 3c, which plots the emission intensity of H0 and H1.5 cell under the applied potentials and clearly shows the relationship between the applied potential against emission quenching for pure NITZ cell (H0) and NITZ/HNI blend cell (H1.5) at two different wavelengths. The cell with NITZ only (H0) showed smaller

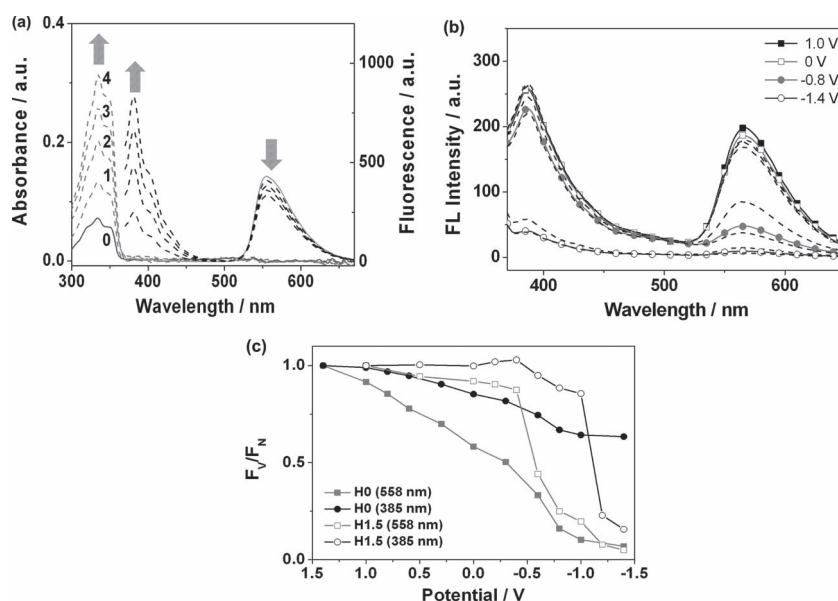


Figure 3. (a) Absorption and fluorescence spectra of NITZ with different content of HNI from bottom to top, 0, 1, 2, 3, 4 eq. (b) Fluorescence changes of a three-electrode switching cell with H1.5 at different applied potentials. Each spectrum was obtained after applying the target potential for 60 s to obtain the fluorescence spectra at saturated state. (c) Relative fluorescence quenching for the two molecular units of TZ (558 nm) and NI (385 nm) under different applied potentials for the cells containing NITZ only (H0) and NITZ blend (H1.5), determined from the fluorescence intensity at given potential (F_v) divided by the fluorescence at neutral states (F_N).

fluorescent quenching (Fq) compared to that with the blend system (H1.5). The H0 cell showed emission quenching only at 558 nm bands as the emission from NI unit (max. at 385 nm) is almost invisible due to the energy transfer from NI to TZ as discussed above. In the blend system (H1.5) the emission from TZ part (max. at 558 nm) started to decrease at -0.5 V and then drastically dropped afterwards. On the other hands, the emission at NI part (385 nm) remained almost the same as that of the neutral molecules between -0.5 V and -1.0 V and then drastically dropped after -1.2 V, which proves that the fluorescence of these cells are controlled by the redox chemistry of NITZ and HNI.

The fact that the emission at NI part (385 nm) remained, while TZ emission is quenched significantly in the applied potential range -0.5 V to -1.0 V, strongly suggests that the occurrence of the yellow fluorescence which is originated from NITZ could be selectively controlled by operating the switching cell at a relatively small potential difference.

The absorption change by applied potential for the 3-electrode NITZ/HNI blend cell (H1.5) was examined with UV-Vis spectroscopy (Figure S2). Upon application of a negative potential below -1.2 V, there was no significant absorption change, while yellow fluorescence was almost extinguished by the reduction of neutral NITZ to their anion-radical form. When the applied potential was below -1.4 V, where reduction of NI unit occurs, the absorption of the H1.5 blend cell (Figure S2b) was increased at visible region showing absorption max. at 408 nm, indicating formation of anion radical of HNI.^[30] On the other hands, there was almost no spectral change for the cell containing only NITZ (H0) (Figure S2a). Thus NITZ is not electrochromic. This indicates that the fluorescence extinction of NITZ could be attributed to the tetrazine unit without contribution from mutual quenching between the excited state of the tetrazine and the already formed NI anion radical through photoinduced electron transfer (PET).^[24] Thus the contribution of the reduced NI unit in NITZ was minor in absorption change, indicating significant energy transfer from NI unit to TZ unit. In the blend system (H1.5), the anion radicals from HNI are in charge of the electrochromism, showing new absorption max. at 408 nm and fluorescence quenching of itself and also neutral fluorophore. The electrochromic and fluorescence switching from the H1.5 cell are compared in Figure S3.

In Figure 2a (and Figure 4a), only yellow fluorescence was switched on and off from NITZ only (H0) by applying negative (-0.8 V \sim -1.4 V) and positive potential (0 V \sim 1.4 V) repeatedly. In the blend cell (H1.5), we observed white emission instead of the yellow emission when the cell was under neutral states (Figure 4a). When both fluorophores emit light, the registered intensity is white, as can be expected from a mixture of the yellow light emitted by NITZ and the blue one from HNI. The white emission was dimmed away and blue emission was observed when the applied potential was in the intermediate range (-0.6 V \sim -1.2 V) and then the fluorescence

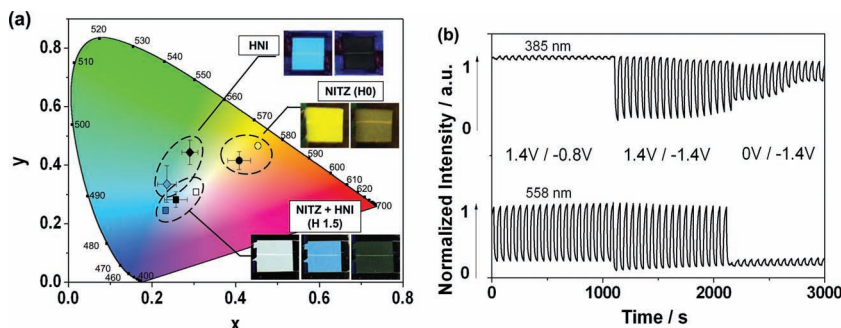


Figure 4. (a) The reversible emission color change of the cell with NITZ blend (H1.5) in chromaticity diagram as compared to the cell of NITZ (H0) and HNI, measured at different potentials. Photographs are the image of the reversible fluorescence switching cells, at given potential. The white bar in the photograph corresponds to a silver wire (1 cm) reference. (b) Fluorescence switching of the blended device (H1.0) between multi-color states. The relative intensity was calculated by dividing FL intensity by initial intensity.

was completely extinguished to dark when the potential was beyond -1.4 V. (Figure 4a). These results can be understood easily based on the redox reaction of each molecules discussed above. As the TZ unit is reduced, the yellow contribution from the TZ units disappeared and only NI emission (blue) remained in the intermediate potential range. Then at the extreme case, where NI reduction occurs at -1.4 V or lower potentials, emission from NI unit is quenched to extinguish fluorescence of the cell. The percentage of the reduced fluorophore was determined from the injected/ejected charge, determined from chronocoulometry for 30 sec at the reduction potential (Figure S4), against calculated charge based on the fluorophore concentration (Supporting information). Upon applying step potential from 1.4 V to -0.8 V, at which only TZ units are reduced, the charge consumption was corresponded to 5.7% of TZ units in NITZ. With a step potential -0.8 V to -1.4 V, at which only NI units are reduced, the charge consumption was corresponded to 4.2% of NI units in NITZ and HNI. With a step potential 1.4 V to -1.4 V, at which both TZ and NI units are reduced, the charge consumption was corresponded to 3.4% of both TZ and NI units (Table S1). Although the conversion from neutral to reduced fluorophore was not completed within 30 sec of reduction time, it was noteworthy that vivid fluorescence switching was achieved, because the sensitivity was high and the switching was driven by the formation of anion radical within electrode diffusion layer.^[24,28] The reduction of TZ unit occurred more than NI unit within white-blue-dark state, because the potential for fluorescence switching was set to reduce TZ unit more drastically than NI unit, based on their cyclic voltammogram, to achieve vivid color change.

Also, when the HNI content was smaller (H0.5), the cell showed pale yellow as the color mixing to reach white emission was incomplete (Figure S5). Importantly the fluorescence from the cell is reasonably switchable at each state, to achieve a multi-color switching electrofluorochromic device. The electrofluorochromic response of the NI part is less reversible than the TZ units, as compared in Figure 4b, possibly due to the unwanted reaction of NI with an avoidable impurities in the far negative potential region. The combination of the NITZ dyad with HNI

for electrofluorochromic cell opens a new method to control fluorescence color.

3. Conclusion

In conclusion, we have demonstrated the reversible electrofluorescence switching of an intrinsically redox active naphthalimide-tetrazine dyad (NITZ) into a multi-time operated device in an all solid state cell in which the fluorescent dyads are in contact with the solid polymer electrolyte. A multi-color electrofluorochromism could be achieved using various types of fluorophores owning at least two stable redox states in a tight cell. The use of a carefully chosen blend of fluorophores and playing on their respective redox potentials, allowed realizing multi-color devices showing respectively the color of the two fluorophores mixed together, then the color of the yet unreacted (neutral) fluorophores, to finish by a completely non-emissive device at very low potentials. Thus by blending NITZ with a naphthalimide (HNI), we obtained white emission at neutral state, blue emission at $-0.8 \sim -1.2$ V, and finally dark at $-1.4 \sim -1.6$ V. The fluorescence from the cell is reasonably switchable at each state, to achieve a multicolor switching electrofluorochromic device.

4. Experimental Section

Materials: Naphthalimide-tetrazine (NITZ) and naphthalimide (HNI) were prepared as previously reported freshly before use.^[27] The materials used for preparing the solid polymer electrolyte (SPE) were methoxy poly(ethylene glycol) monomethacrylate (MPEGM, Mw = 500, Polyscience, Inc.), poly(ethylene glycol) dimethacrylate (PEGDMe, Mn = 550, Aldrich), triallyl-1,3,5-triazine-(1H,3H,5H)-trione (TATT, Aldrich), lithium trifluoromethanesulfonate (LiTFS, Aldrich), and Darocure 1173 (Ciba Geigy). Ag wire used as a reference electrode was purchased from Dasom RMS.

Preparation of Electro-Fluorescent Switching Cells: The fluorescence switching device was made of two layers packed between two transparent ITO electrodes. On one ITO plate, the polymer electrolyte was carefully deposited and cured under UV for 30 min to make a mechanically improved conducting medium. The cured polymer electrolyte solution was initially prepared by mixing MPEGM (0.3 g), PEGDMe (0.6 g), TATT (0.072 g), Darocure 1173 (0.02 g), and LiTFS (0.06 g) according to a method reported in the literature.^[12,15] To a separate polymer batch was added NITZ or HNI and a second ITO plate was coated and used without curing. The NITZ and HNI dissolved easily and NITZ/HNI blend was made by mixing the electrolyte containing 0.5 wt%, 1.0 wt%, and 1.5 wt% of HNI with same volume of the electrolyte containing 1 wt% of NITZ, in this non cross-linked layer. A solid-state switching device was prepared by attaching two ITO glasses with a silver wire between them as a reference electrode. The device was then sealed with epoxy resin. CV was redrawn using ferrocene as an external reference as shown in Figure S6, $E_0(\text{Fc}/\text{Fc}^+) = 0.132$ V.

Measurements: The electrochemical measurements were made on the prepared electro-fluorescent switching cells, and a universal potentiostat (model CHI 624B (CH Instruments, Inc.)). The device fluorescence was measured with luminescence spectrometer- Model LS55 (PerkinElmer). When recording the fluorescence along with the external voltage, the in-situ fluorescence of the switching device was measured using a luminescence spectrometer and the chronocoulometry response was followed accordingly. The CIE colors of emission were measured by Spectroradiometer CS-2000 (Konica Minolta) under UV. The color at each voltage was averaged over 10 cycles of switching. The averaged

values and errors were marked as a points and error bars, respectively, to show average color and reversibility.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

Acknowledgements

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