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Cite this: *Chem. Commun.*, 2020, **56**, 383

Received 9th October 2019,
Accepted 28th November 2019

DOI: 10.1039/c9cc07919j

rsc.li/chemcomm

1,8-Dioxapyrene-based electrofluorochromic supramolecular hyperbranched polymers†

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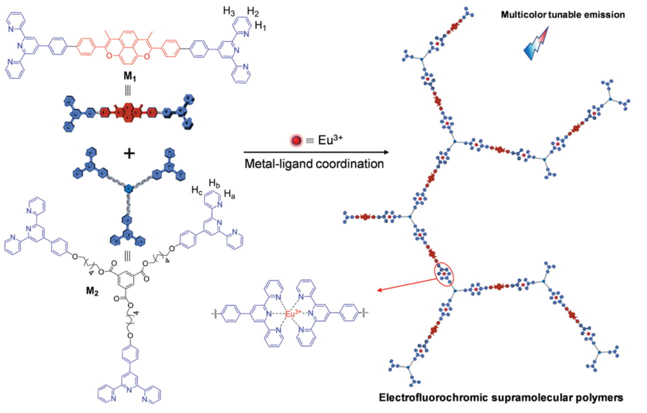
A novel 1,8-dioxapyrene-based electrofluorochromic supramolecular polymer has been successfully constructed, through the coordination of terpyridine ligands with Eu^{3+} ions. The resulting polymer is capable of displaying multicolor tunable capacity and intriguing electrofluorochromic phenomena, and can be used in anti-counterfeiting inks.

Fluorescent supramolecular polymers (FSPs) are drawing great attention in recent years due to their potential in a broad range of applications including optoelectronics,[1] bioimaging,[2] circularly polarized luminescence,[3] and information security.[4] Among them, chromic FSPs are of particular interest, because of their fascinating characteristics in response to defined stimuli, such as heat (thermochromism),[5] light (photochromism),[6] solvent (solvatochromism),[7] vapor (vapochromism),[8] and voltage (electrochromism).[9] In view of the remote and clean stimuli of light and voltage, photochromic and electrochromic FSPs are particularly intriguing and attractive in terms of their broad use in smart materials and molecular devices.[10] Up to now, photochromic FSPs based on photo-responsive molecules such as azobenzene,[11] diarylethene,[12] anthracene[13] and cyanostilbene derivatives have been widely reported.[14] However, electrochromic FSPs have been rarely exploited so far due to a lack of suitable electrochromic units, which can be selected and modified.

Alternatively, 1,8-dioxapyrene and its derivatives have aroused significant interest.[15] These compounds possess remarkable optoelectronic features, and can be recognized as a versatile option for the development of electrochromic FSPs. For the purpose of constructing 1,8-dioxapyrene-based FSPs, an ordinary approach is first to connect assembly elements to the side of such rigid π-conjugated units to form monomers, and then they self-assemble into FSPs.[16] However, the resulting monocomponent FSPs generally cannot meet the requirements of multicolor tunable capacity and multistimuli responsiveness, which restricts their wide range of applications. A superior strategy is to first design 1,8-dioxapyrene-based polypyridine complexes, which can coordinate with various transition metals, and finally to construct multicomponent FSPs through a double monomer polymerization method. Owing to the synergistic participation of 1,8-dioxapyrene fluorophores and transition metal–polypyridine complexes, the multicomponent FSPs exhibit (i) fascinating multicolor tunable features by elaborately regulating the proportion of the two monomers, and (ii) intriguing electrofluorochromic phenomena, which allow for potential applications in the information security field.

Herein, we sought to construct novel electrofluorochromic supramolecular polymers (EFSPs) based on 1,8-dioxapyrene units, through the coordination of terpyridine ligands with Eu^{3+} ions (Scheme 1). Specifically, two different monomers have been first designed and synthesized: the homoditopic building block (**M$_1$**), which contains two symmetrical terpyridine groups on the 2- and 7-positions of the 1,8-dioxapyrene unit, and the homotritopic monomer (**M$_2$**), which consists of three terpyridine



Scheme 1 Schematic illustration of double monomers **M$_1$** and **M$_2$**, and cartoon representation of the resulting EFSPs.

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† Electronic supplementary information (ESI) available: Synthesis, characterization, ^{1}H NMR, spectroscopic data and other materials. See DOI: 10.1039/c9cc07919j

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groups connected by flexible alkyl chains. The synthetic methods and characterization of target compounds are provided in the ESI† (Fig. S1–S15). The metal–ligand coordination among $\mathbf{M_1}$, $\mathbf{M_2}$ and Eu^{3+} ions is expected to produce multicolor tunable EFSPs at appropriate equivalent ratios of $\mathbf{M_1}$ and $\mathbf{M_2}$. Interestingly, the emission colors of the resulting EFSPs can be drastically modulated by voltage or pH, which can be used as fluorescent anti-counterfeiting inks.

Spectroscopic titration experiments were first performed for the confirmation of noncovalent interaction among $\mathbf{M_1}$, $\mathbf{M_2}$, and Eu^{3+} ions. Upon progressive addition of Eu^{3+} ions into the mixed solution of $\mathbf{M_1}$ and $\mathbf{M_2}$ (1 : 1 molar ratio), the emission band at 452 nm gradually decreased in its intensity and concomitantly showed a redshift ($\Delta\lambda$ = 23 nm, Fig. 1a). In addition, the newly emerging bands located at 598 nm ($^5D_0 \rightarrow {}^7F_1$) and 622 nm ($^5D_0 \rightarrow {}^7F_2$) were largely attributed to the intramolecular energy transfer from the excited terpyridine moiety to Eu^{3+} ions.[17] UV-Vis spectra titration experiments also manifested the formation of the complexation of terpyridine groups and Eu^{3+} ions (Fig. 1b). Furthermore, ^1H NMR titration experiments were employed to characterize the formation of EFSPs. Upon mixing $\mathbf{M_1}$ and $\mathbf{M_2}$ with different amounts of $Eu(OTf)_3$ in $CDCl_3$–CD_3CN (1/1, v/v), the aromatic resonances on the terpyridine ligands were significantly broadened and shifted ($\Delta\delta$ = 1.98, 0.86 and 0.35 ppm for $H_{1,2,3}$ on $\mathbf{M_1}$, and 1.97, 1.02 and 0.38 ppm for $H_{a,b,c}$ on $\mathbf{M_2}$, respectively, Fig. 1c). Such phenomena suggest the formation of EFSPs, which can be attributed to the metal–ligand coordination interactions between terpyridine units of $\mathbf{M_1}$ and $\mathbf{M_2}$ upon adding Eu^{3+} ions.

On this basis, we then turn to investigate the emission color variation of the resulting EFSPs by tuning the stoichiometry of $\mathbf{M_1}$ and $\mathbf{M_2}$. Specifically, when the $\mathbf{M_1} : \mathbf{M_2}$ molar ratio is larger than 1 : 10 (*e.g.* $\mathbf{M_1} : \mathbf{M_2}$ = 1 : 5), the main emission band locates at 400–580 nm (Fig. 2a). Meanwhile, the *Commission Internationale de l'Eclairage* (CIE) coordinate is calculated to be (0.28, 0.27), which locates in the blue emission area (Fig. 2b and d). Additionally, when the $\mathbf{M_1} : \mathbf{M_2}$ molar ratio is less than 1 : 10 (*e.g.* $\mathbf{M_1} : \mathbf{M_2}$ = 1 : 20), the emission color of the EFSPs turns to red, and the CIE value is (0.47, 0.30). Intriguingly, a white emission can be observed when the $\mathbf{M_1} : \mathbf{M_2}$ ratio is equal to 1 : 10. The CIE value is calculated to be (0.35, 0.29), which is quite near to the CIE of pure white light (0.33, 0.33) (Fig. 2b and d). The color temperature at this point is noted as 6000 K. In comparison, for the FSPs constructed through the coordination of terpyridine ligands with other transition metal ions (Zn^{2+}, Ln^{3+}, Tb^{3+}, Nd^{3+}, Ni^{2+}, Pr^{3+} and Sc^{3+}), the white-light emission color phenomenon cannot be achieved, because of the single emission band for the resulting EFSPs (Fig. S16, ESI†). Therefore, the emission color of the EFSPs (coordinated with Eu^{3+}) can be tailored from blue to red, and passes through white, by altering the molar ratio of the two monomers $\mathbf{M_1}$ and $\mathbf{M_2}$.

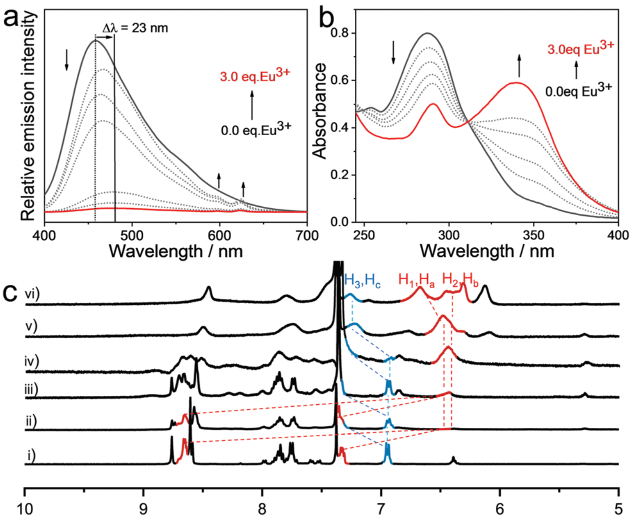

Fig. 1 (a) Fluorescence and (b) UV-Vis spectra change upon gradual addition of Eu^{3+} into the mixture solution of $\mathbf{M_1}$ and $\mathbf{M_2}$ (1 : 1 molar ratio, $CHCl_3/CH_3CN$ = 1 : 1 v/v, 0.01 mM). λ_{ex} = 340 nm. (c) Partial ^1H NMR spectra (400 MHz, $CDCl_3/CD_3CN$ (1 : 1, v/v), 298 K, 5 mM) of the mixture of $\mathbf{M_1}$, $\mathbf{M_2}$ and $Eu(OTf)_3$ at different molar ratios, (i) 1 : 1 : 0, (ii) 1 : 1 : 0.5, (iii) 1 : 1 : 1, (iv) 1 : 1 : 1.5, (v) 1 : 1 : 2, and (vi) 1 : 1 : 2.5.

After developing white-light EFSPs, we then turn to gain further insight into the luminescence properties of this polymer. In this regard, the time-resolved fluorescence decay profile of the white-light EFSPs is fitted as a single exponential decay with a lifetime of 902 μs (Fig. 2c). In the meantime, the fluorescence quantum yield was measured to be 0.33 (Table S1, ESI†). Hence, by precisely regulating the ratio of the 1,8-dioxapyrene fluorophores and terpyridine–Eu^{3+} complexes, the long-lived white phosphorescent supramolecular polymer can be successfully exploited.

As mentioned in the literature, 1,8-dioxapyrene could undergo a remarkable redox process when voltage is applied.[15b] Thus, *in situ* UV-Vis spectra electrochemical measurements were first performed for monomer $\mathbf{M_1}$. When applying a positive voltage of +1.5 V, a novel absorption band appears at 482 nm, while the

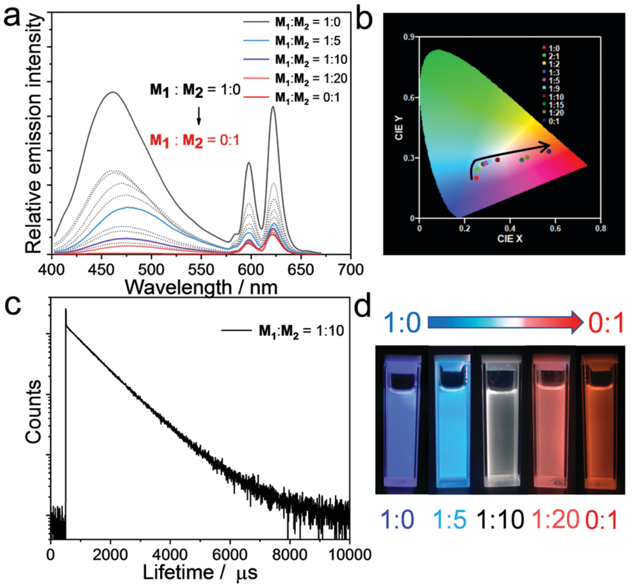

Fig. 2 (a) Fluorescence spectra and (b) the CIE chromaticity diagram of the EFSPs with different molar ratios of $\mathbf{M_1}$ and $\mathbf{M_2}$ in $CHCl_3/CH_3CN$ (1 : 1, v/v) at λ_{ex} = 340 nm. (c) Fluorescence decay profiles of the EFSPs ($\mathbf{M_1} : \mathbf{M_2}$ = 1 : 10). (d) Selected fluorescence photographs of EFSP solutions with different molar ratios of $\mathbf{M_1}$ and $\mathbf{M_2}$.

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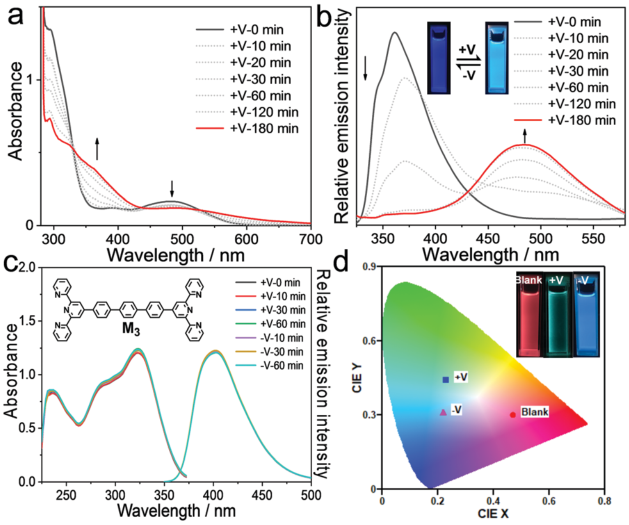

Fig. 3 UV-Vis and fluorescence spectra variations of (a and b) $\mathbf{M_1}$ and (c) $\mathbf{M_3}$ upon continuously applying a voltage. Inset of (b): the emission color of $\mathbf{M_1}$ upon positive and negative voltage treatments. (d) The CIE chromaticity diagram of the EFSPs ($\mathbf{M_1:M_2}$ = 1:20) before and after applying the positive and negative voltages, respectively. The concentrations in (a)–(c) are 0.01 M in $CHCl_3$/MeCN = 1:1, v/v with 0.1 M tetrabutyl ammonium hexafluorophosphate as electrolyte.

band centered at 294 nm gradually decreases, with three isosbestic points at 330, 438, and 529 nm (Fig. 3a). Simultaneously, as the energization time is continued, the emission band (λ_{max} = 366 nm) of $\mathbf{M_1}$ gradually red-shifts to 485 nm (Fig. 3b). In addition, emission color changes from blue to cyan are observed after applying the positive voltage to $\mathbf{M_1}$ (Fig. 3b, inset). Such phenomena reveal the conversion from $\mathbf{M_1}$ to its electrochemical products. The process can be further demonstrated by means of ^{1}H NMR experiments (Fig. S18, ESI†), which show the disappearance of 1,8-dioxapyrene resonances ($H_{a'}$ 6.47 ppm, $H_{b'}$ 6.44 ppm), together with the emergence of a new set of resonances in the downfield region ($H_{a'}$ 7.06 ppm, $H_{b'}$ 7.03 ppm). When the solution of $\mathbf{M_1}$ is subjected to a negative voltage of −1.5 V, the UV-Vis and emission spectra are completely recovered, together with the return of emission color to being blue (Fig. S19, ESI†). As a control experiment, when positive or negative potential is exerted on the control compound $\mathbf{M_3}$ (see the structure in Fig. 3c, inset), both UV-Vis and fluorescence spectroscopic signals hardly change (Fig. 3c). Therefore, we rationalized that the 1,8-dioxapyrene unit could undergo redox reactions as a function of the applied potential, which leads to the appearance of electrofluorochromic features.

Benefiting from the excellent photoelectric properties of monomer $\mathbf{M_1}$, we then turn to investigate the nature of the EFSPs. When a +1.5 V voltage is incessantly applied to the solution of the red emission supramolecular polymer ($\mathbf{M_1:M_2}$ = 1:20; CIE coordinate is (0.47, 0.30)), the emission band at 472 nm gradually red-shifts to 505 nm, together with enhanced intensity (Fig. S20a, ESI†). During this process, the fluorescence color is gradually dominated by the band at 505 nm, which changes from red to green (CIE coordinate is (0.23, 0.44), Fig. 3d). Furthermore, when the positive voltage is converted to a negative one, the emission band at 504 nm blue-shifts to 481 nm, and the intensity gradually decreases as the energization time is extended (Fig. S20b, ESI†). The emission color of the solution varies from green to blue (CIE coordinate is (0.22, 0.31), Fig. 3d).

It is worth noting that the coordination bonds of terpyridine–Eu^{3+} also play an important role in regulating the emission color of the EFSPs. Specifically, the counterpart EFSPs ($\mathbf{M_3:M_2}$ = 1:20) were prepared under the same conditions. During the electrification process, the UV-Vis spectroscopic signals scarcely change, indicating that the coordination bond cannot be destroyed by electricity (Fig. S21a, ESI†). Interestingly, the emission band (λ_{max} = 485 nm) of the counterpart EFSPs gradually increases under energized conditions, which can regulate the relative intensity between blue and red emissions (Fig. S21b, ESI†). Accordingly, by the synergistic participation of 1,8-dioxapyrene units and terpyridine–Eu^{3+} complexes, the emission signals of the EFSPs can be fine-tuned by electrical stimuli, which possess the remarkable electrofluorochromic properties.

We then sought to exploit fluorescent anti-counterfeiting applications of the EFSPs, by taking full advantage of the emission color variation regulated by voltage or pH. Specifically, the EFSP solution ($\mathbf{M_1:M_2}$ = 1:20) is loaded into a fountain pen, and a word "NPU" can be written on non-fluorescent paper, which is invisible under natural light and shows red emission color under UV light (Fig. 4a). When the paper is fumed with trifluoroacetic acid (TFA) vapor, the emission color of "NPU" promptly turns to green. Subsequently, the red emission of "NPU" could be fully restored when the paper is fumed with trimethylamine (TEA) vapor. The phenomena are highly plausible, since the redox reactions of $\mathbf{M_1}$ can also be triggered by pH. With the successive TFA and TEA vapor treatments, the emission colors of the word can be reversibly switched between red and green for multiple cycles (Fig. 4b). Considering the unique electrofluorochromic feature of the EFSPs, a colorful cartoon pattern is painted on a paper by using three different emission colors of EFSPs as anti-counterfeiting inks (Fig. 4c). A liquid optoelectronic device is

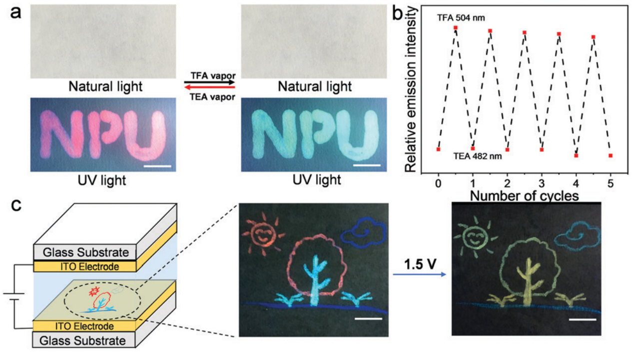

Fig. 4 (a) Photographs of the word "NPU" after fuming with TFA and TEA vapor under natural light and 365 nm UV light. Scale bar: 1 cm. (b) Relative fluorescence intensity of the word "NPU" as a function of the TFA–TEA cycle numbers. The fluorescence intensity was monitored at wavelengths of 504 and 482 nm, respectively. (c) Schematic representation of the electrochromic device and photographs of the cartoon pattern by painting with three different EFSP inks (red emission: $\mathbf{M_1:M_2}$ = 1:20, cyan emission: $\mathbf{M_1:M_2}$ = 1:5, and blue emission: $\mathbf{M_1:M_2}$ = 2:1) on the paper. Scale bar: 1 cm.

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fabricated by encapsulating the pattern into ammonium hexafluorophosphate conductive media and sandwiching between two indium-tin oxide (ITO) anodes. When a positive voltage is applied to the device, the pattern shows evident emission color change in a few seconds (Fig. 4c).

In summary, we have successfully constructed new electrofluorochromic supramolecular polymers (EFSPs) based on 1,8-dioxapyrene units through the coordination of terpyridine ligands with Eu^{3+} ions. By controllably adjusting the ratio of monomers $\mathbf{M_1}$ and $\mathbf{M_2}$, the resulting EFSPs exhibit multicolor emission features from blue to red, passing through white. Thanks to the synergistic participation of 1,8-dioxapyrene and terpyridine–Eu^{3+} complexes, the emission signals of the EFSPs can be fine-tuned by electrical stimuli, which possess remarkable electrofluorochromic properties. Finally, the EFSPs feature distinct emission colors upon voltage or pH treatment, which can be employed for fluorescent anti-counterfeiting applications with high security. Hence, EFSPs with multicolor tunable capacity will be promising candidates for application in information security protection technology.

We would like to acknowledge the support of the National Science Foundation of China (No. 21674086), the Natural Science Basic Research Plan in Shaanxi Province of China (2018JZ2003), and the Fundamental Research Funds for the Central Universities (3102019PY003 and G2019KY05111). We also thank the Analytical & Testing Center of Northwestern Polytechnical University for electron microscope tests.

Conflicts of interest

There are no conflicts to declare.

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