



The Flexible Electrochromic Device That Turns Blue Under Both Positive and Negative Application Voltages

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Using the photocurable electrolyte solution containing viologen derivative 1,1'-bis-(4-vinylbenzyl)-4,4'-bipyridinium dihexafluorophosphate ($EV^{2+} \bullet 2PF_6^-$) and its composite of $EV^{2+} \bullet 2PF_6^-$ and triphenylamine derivative *N,N'*-di(4-((3-(triethoxysilyl)propyl)imino)methyl)phenyl-*N,N'*-diphenyl-4,4'-biphenyldiamine (TPB-PSSO) as the electrochromic active layer, respectively, the flexible electrochromic devices (FECDs) were prepared by photocurable technology. The device structure was PET-ITO/photocurable electrolyte solution/PET-ITO. The electrochromic properties of FECDs were investigated. The results show that the FECDs can all undergo a reversible color change, especially the PET-ITO/photocurable electrolyte solution-composite/PET-ITO can undergo polychromatic transition. The FECD based on $EV^{2+} \bullet 2PF_6^-$ can reversibly change between colorless and deep blue, and the FECD based on the composite of TPB-PSSO- $EV^{2+} \bullet 2PF_6^-$ can reversibly change between black blue, light yellow, and sky blue.

Keywords: viologen derivative, triphenylamine derivative, composite, photocurable electrolyte solution, flexible electrochromic devices

1 INTRODUCTION

In recent years, people have paid more and more attention to energy-saving, which means electrochromic materials and corresponding devices have gradually become a research hotspot. The organic molecules can undergo a reversible electrochemical redox under an applied voltage, resulting in a color change known as organic electrochromic materials. Organic electrochromic materials have been studied because of their advantages, such as easy synthesis, modification, and fabrication of flexible devices (Liu et al., 2020a; Liu et al., 2020b; Frolov et al., 2020; Ma et al., 2020; Nad and Malik, 2020; Xiao et al., 2020; Zhang et al., 2020; Kim et al., 2021; Zhang X et al., 2021; Kim et al., 2021; Hao et al., 2021; Huang et al., 2021; Li et al., 2021). Organic electrochromic devices (ECDs) have good application prospects, such as smart windows and displays. In our previous work, a series of triphenylamine (TPA) derivatives were synthesized, and their electrochromic properties were investigated (Liu et al., 2017; Zeng et al., 2017; Zeng et al., 2018; Zeng et al., 2019). As solid films, these derivatives exhibited high optical contrast and a short response time under applied voltages. In addition, a series of conjugated linear and star oligothiophene derivatives and organic conjugated oligomers containing 4-ethylenedioxythiophene were synthesized (La et al., 2008; Yin et al., 2010; Jiang et al., 2012; Zhong et al., 2015; Wan et al., 2018). These organic compounds exhibited reversible and distinct chromatic changes. 4,4-Bipyridine, the core group of viologen derivatives, has a strong electron-acquiring ability. The N atom of TPA derivatives has a lone electron-pair and strong electron-supplying ability, making them prone to electron gain and loss.

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In this study, the *N,N'*-di(4-((3-(triethoxysilyl)propyl) imino)methyl)phenyl-*N,N'*-diphenyl-4,4'-biphenyldiamine (TPB-PSSO) and a 1,1'-bis(4-vinylbenzyl)-4,4'-bipyridinium dihexafluorophosphate ($\text{EV}^{2+}\bullet 2\text{PF}_6^-$) were synthesized. Using the photocurable electrolyte solution containing $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ and PSSO- $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ composite as the electrochromic active layer, respectively, the flexible electrochromic devices (FECDs) were prepared, and their electrochromic properties were investigated. The molecular structures of TPB-PSSO and $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ are presented below (Figure 1).

2 EXPERIMENTAL

2.1 Materials

All solvents were purified and dried using standard methods. Triphenylamine (TPA), phosphorus oxychloride (POCl_3), NH_4PF_6 , photocurable resin (PET-400DA), and photoinitiator-184 were purchased from Alfa Aesar Chemical Co., Ltd. Lithium perchlorate (LiClO_4), tetrahydrofuran (THF), and potassium carbonate (K_2CO_3) were purchased from Shanghai RichJoint Chemical Reagents Co., Ltd. 1,2-Dichloroethane, acetonitrile (ACN), and ethanol (EtOH) were purchased from Tianjin Fuyu Fine Chemical Co. Ltd. *N,N*-Dimethylformamide (DMF) and nitric acid (HNO_3 , 65%) were purchased from Guangzhou Chemical Reagent Factory. Sodium hydroxide (NaOH) was purchased from Fuchen (Tianjin) Chemical Reagents Co., Ltd. 3-Aminopropyl triethoxysilane (APTES) was purchased from Foshan Daoning Chemicals Ltd. Dichloromethane (DCM) and propylene carbonate (PC) were obtained from Sinopharm Chemical Reagent Co., Ltd. Indium tin oxide (ITO) PET (10 Ω) was purchased from Zhuhai Kaivo Optoelectronic Technology Co., Ltd.

2.2 Instruments

Fourier transform infrared (FTIR) spectroscopy was performed on a Nicolet 6700 (Thermo Fisher Scientific) spectrometer. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AVANCE-600 NMR spectrometer. Using a Thermo Helios-g spectrometer, the ultraviolet-visible (UV-vis) spectra were recorded. Using a CHI750A electrochemical workstation (Zhenhua Apparatus Co., Shanghai), the electrochemical properties were measured in a PC solution containing 0.1 M LiClO_4 . A saturated calomel electrode (SCE), platinum wire, and glassy carbon electrode were used as the reference electrode (RE), counter electrode (CE), and working electrode (WE), respectively.

2.3 Synthesis

2.3.1 Synthesis of *N,N'*-Di(4-((3-(triethoxysilyl)propyl) imino)methyl)phenyl-*N,N'*-diphenyl-4,4'-biphenyldiamine (TPB-PSSO)

The TPB-PSSO was synthesized and characterized according to the procedure reported (Zhang X. et al., 2021).

2.3.2 Synthesis of

1,1'-Bis(4-vinylbenzyl)-4,4'-bipyridinium Dihexafluorophosphate ($\text{EV}^{2+}\bullet 2\text{PF}_6^-$)

4,4'-Bipyridine of 3.12 g (20 mmol), 60 ml acetonitrile (ACN), and 5 ml (60 mmol) ethane bromide were added into a 100 ml single flask. The solution was stirred under N_2 at 80°C for 48 h. Then, the solution was filtrated, and a yellowish crude product was obtained. The yellowish crude product was washed with ACN and dried in a vacuum. The 30 ml NH_4PF_6 aqueous solution was added to the aqueous solution of the above yellow product, and the mixture solution was stirred quickly for 3 h. Then, the mixture solution was filtrated, and a white crude product was obtained. The white crude product was washed with distilled water and vacuum drying. The 9.3 g white $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ powder was obtained. ^1H NMR (DMSO, 400 MHz, ppm): 9.38 (d, $J = 7.2$ Hz, 4H), 8.76 (d, $J = 6.9$ Hz, 4H), 4.73 (m, $J = 2.0$ Hz, 4H), 1.61 (t, $J = 2.0$ Hz, 6H). ^{13}C -NMR (DMSO, 400 MHz, ppm): 149.10, 145.96, 127.07, 57.07, 16.85; HRMS (ESI): ($\text{C}_{14}\text{H}_{18}\text{N}_2^+$) 214.1417, calcd. for 214.1439.

2.4 Preparation of the Photocurable Electrolyte Solution

The LiClO_4 and $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ [or composite of TPB-PSSO- $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ (w/w = 1/1)] were added to 25 ml of propylene carbonate (PC). The solution was stirred until completely dissolved. The concentration of LiClO_4 is 2.83 mol/L, and the concentration of $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ [or composite of TPB-PSSO- $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ ((w/w = 1/1))] is 0.01 M. Then, 0.80 g of photocurable resin (PET-400DA) and 0.02 g of photoinitiator 184 were added to the abovementioned electrolyte solution and stirred evenly. The prepared photocurable electrolyte was stored in an N_2 atmosphere away from light, hence being ready for use.

2.5 Preparation of the Flexible Electrochromic Devices

The FECDs with the sandwich structure were assembled using the two pieces of $2.5 \times 5 \text{ cm}^2$ ITO-PET substrate as the electrode. A certain amount of the abovementioned two-electrolyte solution was injected into the hollow part of the sandwich structure, respectively. Then, the FECDs were put into the photocuring box for 1 min. The structure of FECDs was PET-ITO/photocurable electrolyte solution/PET-ITO. The FECDs based on the $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ and the composite of TPB-PSSO/ $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ (W/W = 1:1) were named FECD-EP and FECD-composite, respectively.

3 RESULTS AND DISCUSSION

3.1 Optoelectrochemical Properties of TPB-PSSO and $\text{EV}^{2+}\bullet 2\text{PF}_6^-$

Figure 2 shows the UV-vis spectra of TPB-PSSO and $\text{EV}^{2+}\bullet 2\text{PF}_6^-$ in PC solution, respectively. The maximum absorption wavelength (λ_{max}) of TPB-PSSO PC solution is 362 nm; that

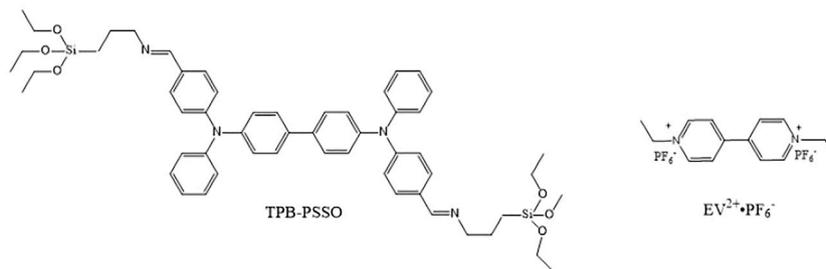


FIGURE 1 | The molecular structure of TPB-PSSO and EV²⁺•2PF₆⁻.

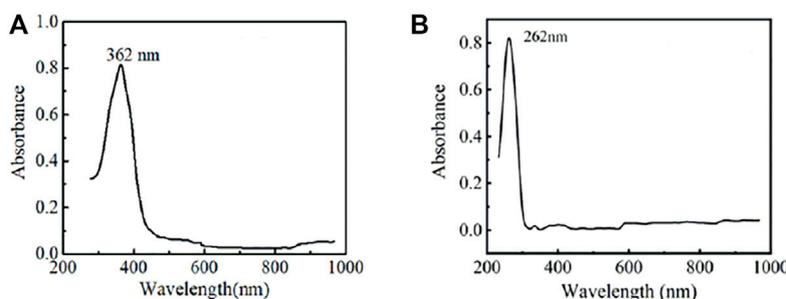


FIGURE 2 | The UV-vis spectra of TPB-PSSO (A) and EV²⁺•2PF₆⁻ (B).

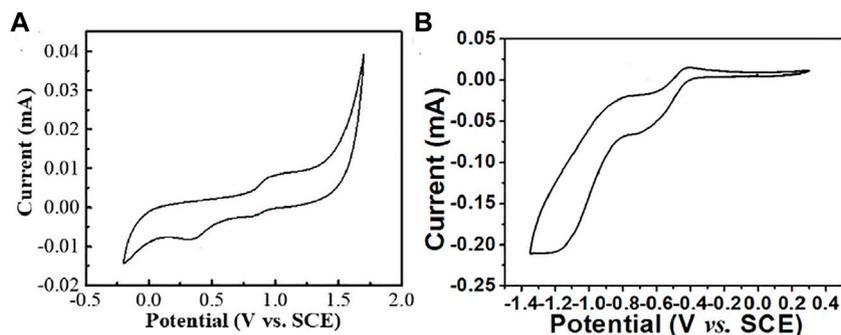


FIGURE 3 | The CV curve of TPB-PSSO (A) and EV²⁺•2PF₆⁻ (B).

TABLE 1 | The electrochemical redox ability of compounds.

Compounds	E _{ox} ^{onset} (V vs. SCE)	E _{red} ^{onset} (V vs. SCE)
TPB-PSSO	0.80	-0.15
EV ²⁺ •2PF ₆ ⁻	—	-0.42

is, the color of the TPB-PSSO is yellow. The maximum absorption wavelength (λ_{max}) of EV²⁺•2PF₆⁻ in the PC solution is 262 nm, and there is no obvious absorption peak in the visible region, which shows that the EV²⁺•2PF₆⁻ solution is colorless and transparent.

The electrochemical redox ability of organic compounds was tested by cyclic voltammograms (CV). The scanning was carried out at a rate of 50 mV/s. As shown in **Figure 3** and **Table 1**, the onset of oxidation and reduction potential of TPB-PSSO were 0.80 and -0.15 V (vs. SCE), respectively. The onset of reduction potential of EV²⁺•2PF₆⁻ was -0.42 V (vs. SCE). The CV results show that these two compounds have oxidation and reduction abilities and may be used as electrochromic active material.

3.2 Electrochromic Properties of the FECDs

Figure 4 shows the CV curves of FECD-EP and FECD-composite, respectively. The reversible redox process of

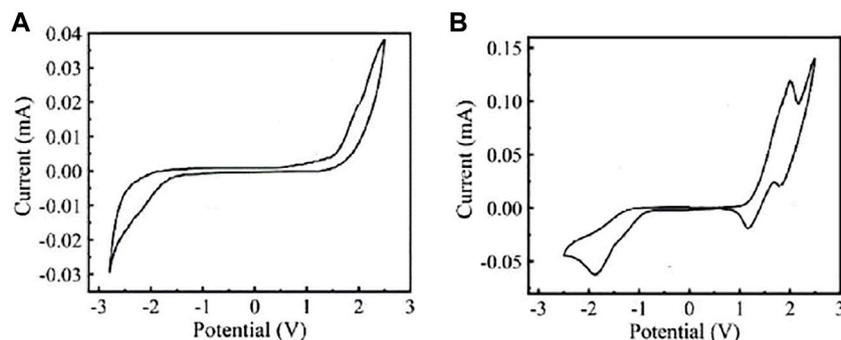


FIGURE 4 | The CV curve of FECD-EP (A) and FECD-composite (B).

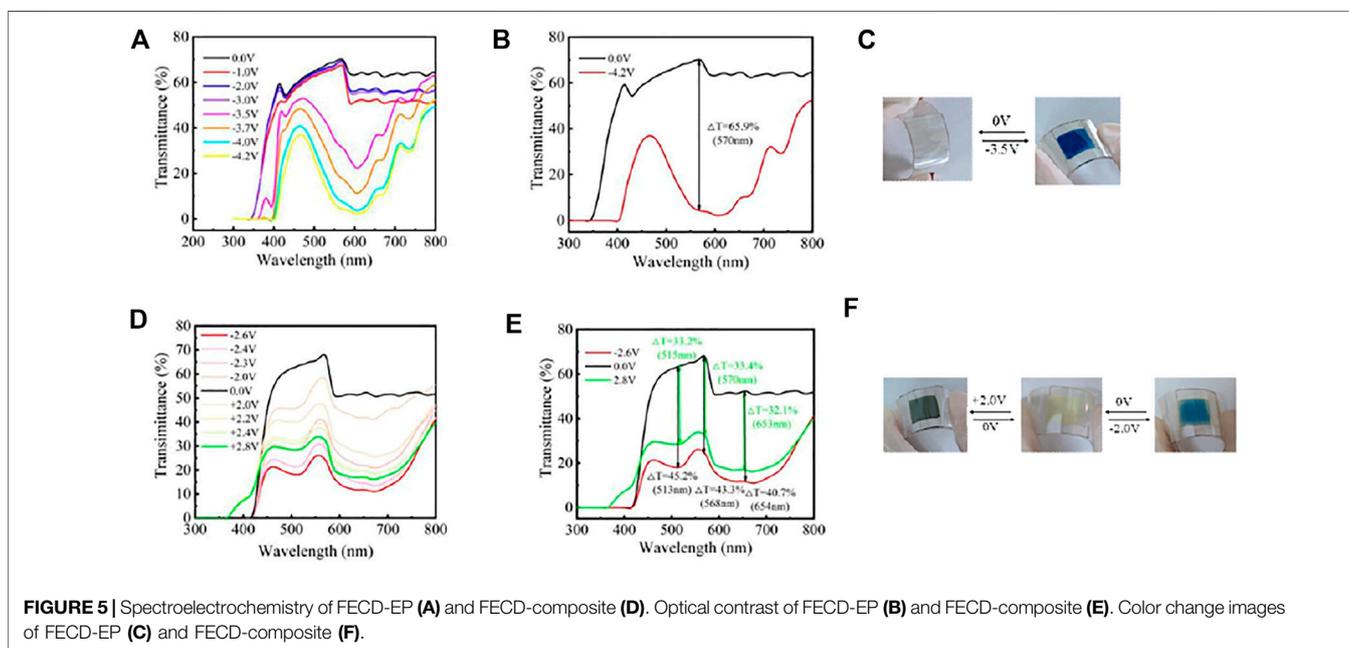


FIGURE 5 | Spectroelectrochemistry of FECD-EP (A) and FECD-composite (D). Optical contrast of FECD-EP (B) and FECD-composite (E). Color change images of FECD-EP (C) and FECD-composite (F).

FECD-EP is the conversion between the EV^{2+} and radical $EV^{\bullet+}$ accompanied by the gain and loss of an electron. The FECD-composite has two reversible redox processes: one is the reversible redox process of TPB-PSSO and the other is the reversible redox process of $EV^{2+} \bullet 2PF_6^-$.

As shown in **Figure 5**, the FECD-EP and FECD-composite can undergo reversible color change under applied voltage. When the applied voltage is at 0 V, the FECD-EP is colorless, and when the applied voltage is at -3.5 V, the color of the FECD-EP is deep blue. The main reason for this color change is that the EV^{2+} can get an electron under the applied voltage and be reduced to the monovalent cation radical $EV^{\bullet+}$. When the applied voltage is 0 V, the FECD-composite is light yellow. When the applied voltage is at 2.0 V, the color of the FECD-composite is black blue. When the applied voltage is at -2.0 V, the color of FECD-composite is sky blue.

Optical contrast (ΔT) refers to the difference in the optical transmittance of ECD in coloring and bleaching state at a certain wavelength. The ΔT of FECD-EP was 65.9% at 570 nm. The ΔT of the FECD-composite was 33.2%, 33.4%, and 32.1% at 515, 570, and 653 nm, respectively.

The response time of FECD-EP and FECD-composite is measured by chronoamperometry. The time required for the current change to reach 95% during coloring and bleaching is denoted as coloring response time (t_c) and bleaching response time (t_b), respectively. The timing-current spectra of FECD-EP and FECD-composite are shown in **Figure 6**. When FECD-EP alternatively switches the voltage of -3.0 and 1.0 V, the T_c is 0.2 s and T_b is 0.1 s. When the applied voltage of -2.5 and 2.5 V is alternately switched with FECD-composite, the t_c and t_b are 1.8 and 4.4 s. Comparing the t_c and t_b of FECD-EP, it can be found that the coloring and bleaching time of the FECD-composite is longer.

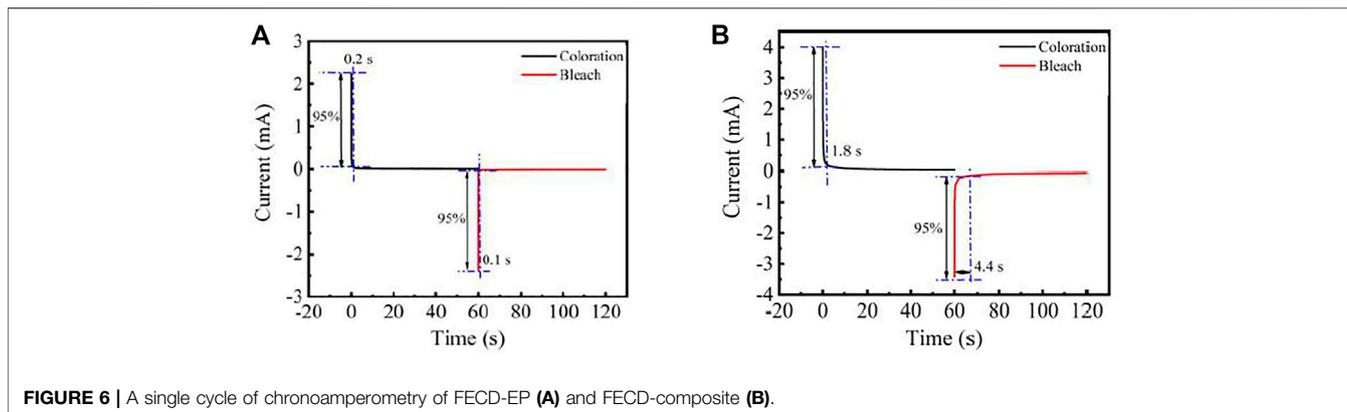


FIGURE 6 | A single cycle of chronoamperometry of FECD-EP (A) and FECD-composite (B).

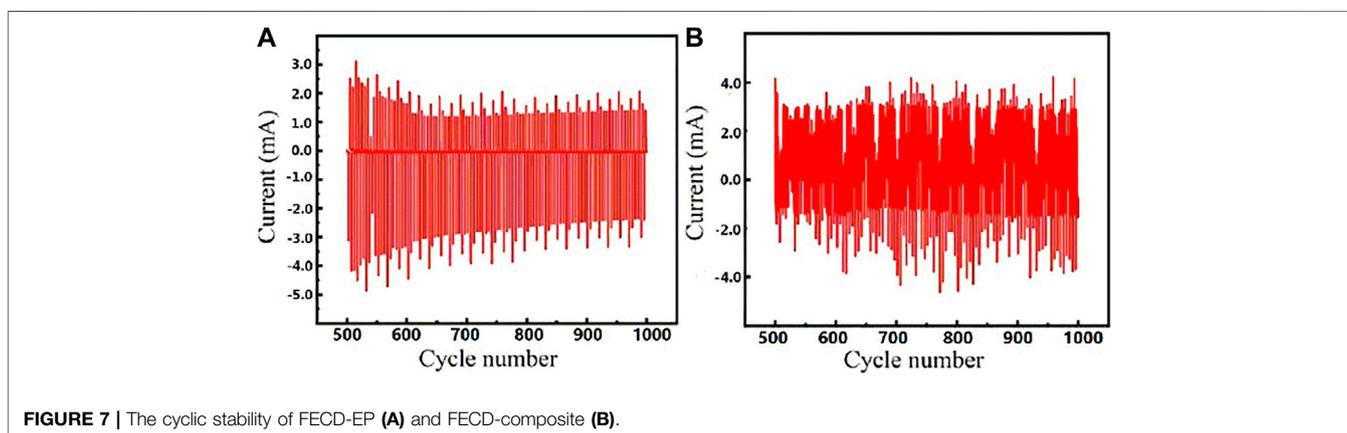


FIGURE 7 | The cyclic stability of FECD-EP (A) and FECD-composite (B).

Figure 7 shows the cyclic stability curves of FECD-EP and FECD-composite. The FECD-EP retained 67.1% of their original charge after 1,000 cycles. However, the timing-current curve of the FECD-composite fluctuated greatly, and the amount of charge at the 1,000th cycle was slightly higher than the initial charge. The reason might be that excessively high driving voltage may easily cause over-oxidation or over-reduction of electrochromic materials in the electrochromic process.

The coloration efficiency of an electrochromic device refers to the change value of the optical density of the device when the unit charge is injected or extracted during the coloration process. The coloration efficiency of FECD-EP and FECD-composite can be calculated according to Eq. (1):

$$\eta = \Delta OD / Q, \Delta OD = \log(T_b / T_c). \quad (1)$$

The η is the coloration efficiency (cm^2/C), ΔOD is the change of optical density, Q is the amount of charge injected (or withdrawn) (cm^2/C) of unit area, and T_c and T_b are, respectively, the optical transmittance of colored state (%) and bleached state (%) at a specific wavelength. The η of FECD-EP at 570 nm was $151.50 \text{ cm}^2/\text{C}$. When the positive applied voltage was applied, the η of FECD-composite at 515, 570, and 653 nm was $74.15 \text{ cm}^2/\text{C}$, $68.16 \text{ cm}^2/\text{C}$, and $104.86 \text{ cm}^2/\text{C}$, respectively. When

the reverse applied voltage was applied, the η of FECD-composite at 513, 568, and 654 nm was $116.70 \text{ cm}^2/\text{C}$, $93.25 \text{ cm}^2/\text{C}$, and $140.28 \text{ cm}^2/\text{C}$.

4 CONCLUSION

Using the photocurable electrolyte solution containing $\text{EV}2^+ \bullet 2\text{PF}_6^-$ and its composite of $\text{EV}2^+ \bullet 2\text{PF}_6^-$ and TPB-PSSO as the electrochromic active layer, respectively, the FECDs were prepared by photocurable technology. The FECD based on $\text{EV}2^+ \bullet 2\text{PF}_6^-$ can reversibly change between colorless transparent and deep blue, and the FECD based on the composite of TPB-PSSO- $\text{EV}2^+ \bullet 2\text{PF}_6^-$ can reversibly change from light yellow to sky blue and dark blue under both positive and negative application voltages.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusion of this article will be made available by the authors without undue reservation.

AUTHOR CONTRIBUTIONS

SJ: methodology, formal analysis, investigation, writing—original draft. XZ: validation. XG: conceptualization, software, data curation. MZ: visualization. PL: resources, writing—review and editing, supervision, project administration, funding acquisition.

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