ABSTRACT: Lithium-ion-assisted ultrafast charging double-electrode smart windows with energy storage and a fluorescence display device (FTO/PB/Ru@SiO2||Ru@SiO2/WO/FTO) based on double electrochromic electrodes (cathode and anode) (FSDECEs) have been designed and fabricated. Here, Prussian blue (PB) and WO_{red} are selected as the electrochromic cathode and anode, respectively. There is a synergistic effect and a large potential difference between the two electrodes. They could be simultaneously and rapidly bleached after being connected with each other. Also, the fluorescence intensity of Ru@SiO2 nanoparticles (NPs) could be regulated by the fluorescence resonance energy transfer effect (FRET). After discharging, the two electrochromic electrodes in the bleached state can be recharged by a Mg−O_{2} battery with a FeN₅ single atomic catalyst to quickly recover the colored state. The double electrochromic electrodes can reversibly alter between coloring and bleaching states only by connecting and disconnecting the electrodes. The fluorescence intensity of FSDECEs can switch between quenching and emission, thus endowing the “on” and “off” functions. The system is concise, environmentally friendly, and easy to operate. The proposed FSDECEs demonstrate high fluorescence contrast, a fast response time, and long-term stability. Such an ingenious design of fluorescence switching based on the double electrochromic electrode in a single cell sheds light on next-generation transparent, portable, and self-powered electrochromic devices and electronic equipment.

INTRODUCTION

Recently, a transparent electrode has attained considerable attention because it is a key component in next-generation transparent optical electronic devices, such as solar cells, touch screens, and healthcare sensors. A reversible change in the oxidation/reduction states of the electrochromic materials is followed along with the variation of the electrode color and absorbance, which could be widely applied in various electrochromic materials. Prussian blue (PB) as a common electrochromic material has been favored by researchers for its excellent electrochromic properties. It can be reduced to Prussian white (PW) under electrical stimulation and takes on bleaching states. It is a suitable material for preparing transparent electrochromic batteries. In recent years, there have been many reports on electrochromic electrodes based on PB or PB analogues. However, conventional electrochromic batteries usually have only single electrode with electrochromic material. Actually, the battery containing double electrodes (anode and cathode) with electrochromic materials is more convenient and efficient at storing and releasing energy when needed in transparent portable electronic device, so it is of great significance to study a battery with double electrochromic electrode materials. At present, batteries containing both an anode and cathode with electrochromic materials are rather rare. And the processes of charging and discharging an electrochromic battery usually depends on the external power source, which causes a waste of energy and increases the complexity of the system to some extent. As we know, electron transfer could spontaneously occur between electrodes with materials possessing a potential difference when they are connected. This is a thermodynamically spontaneous process (ΔG < 0). It is particularly important to select two suitable electrochromic materials. The alternative double electrochromic materials should meet the following two requirements: (1) The potentials of the double electrochromic electrodes should be matched well, namely, one is an anode electrochromic material and the other is a cathode electrochromic material. (2) They can be colored or bleached simultaneously under the same potential. Coincidentally, tungsten oxide (WO) is another popular electrochromic material, and it can be reduced to its reduction form WO_{red} (M(WO_{3−α}), M = H⁺, Li⁺, Na⁺) under the proper stimulation. More importantly, its redox potential matches the PB potential well, so WO is chosen as another electrochromic material to match PB in order to construct a double electrochromic battery.
In addition, there is a synergistic effect between PB and WO electrodes, which is good for expediting the electrochromic process. Furthermore, consider that both materials possess feasible structures for the transport of positive ions, which favors energy storage. In particular, the small radius of the Li\(^+\) ion could rapidly insert in and exit from the PB and WO host, which further accelerates the time of the electrochromic process of PB and WO, and a fast response is beneficial to practical applications.

PB (coloring) could oxidize WO\(_{\text{red}}\) (coloring) when the two electrodes are connected, and the two colored electrodes could quickly be bleached at the same time. However, one formidable challenge is the slow recoloring of the electrodes after bleaching, especially for the oxidation of PW to PB. A usual way to solve this problem is by adding some oxidizing agents to the system to speed up the reaction, but it causes the system to be complex and unstable. More crucially, oxidizing agents can promote the oxidation of PW to PB, but they can inhibit the reduction of WO. Therefore, the approach of adding oxidizing agents to one cell is not feasible in our proposed system. To solve the synchronous coloring problem of the two electrodes after discharge, a Mg\(_2\)O\(_2\) battery is employed to charge the FTO/PB|\|WO\(_{\text{red}}\)/FTO double electrochromic electrodes after discharge. A FeN\(_5\) single atomic catalyst is employed in the proposed system to catalyze the O\(_2\) reduction reaction (ORR) process with high catalytic activity but more cheaply than can Pt/C. The introduction of the Mg–O\(_2\) battery works out the slow recoloring problem of the two bleaching electrodes and greatly shortens the time of coloring/charging.

Fluorescence switching is an interesting stimulus-responsive fluorescence display system, and its fluorescence intensity can be regulated through fluorescence resonance energy transfer (FRET) between donors (fluorescence molecule) and acceptors (electrochromic materials). The reported fluorescence switching based on electrochromic electrodes is limited by a single electrochromic electrode system. However, double electrochromic electrode fluorescence switching can store and release more energy when needed, and it can show higher fluorescence intensity at the same time. The fluorescence contrast can be enhanced notably compared to that of single electrode fluorescence switching. In addition, the sensitivity of the fluorescence switching can be improved markedly when combining electrochemistry with fluorescence spectroscopy. The two electrochromic electrodes can simultaneously act on the intermediate fluorescent molecules, greatly improving the fluorescence contrast, especially compared to single electrode fluorescence switches. As far as we know, this is the first example of reported FSDECEs.

Herein, the FSDECEs of FTO/PB/Ru@SiO\(_2\)\|\|Ru@SiO\(_2\)/WO/FTO with both an anode and cathode as electrochromic electrodes have been put forward (Scheme 1). First, the WO\(_{\text{red}}\) electrode (coloring) and PB electrode (coloring) are assembled in one cell as the cathode and anode of FSDECEs, respectively. The fluorescence is quenched by the FRET of the donor (Ru@SiO\(_2\)) and acceptor (WO\(_{\text{red}}\) and PB), denoted as the off state. Then the two coloring electrochromic electrodes are connected together and become a bleaching state simultaneously (Figure S1). At this time, the fluorescence of FSDECEs cannot be quenched due to the very weak absorbance of PW and WO electrodes, marked as the on state. Immediately, the bleaching PW and WO electrode are connected with FeN\(_5\) and the metal Mg electrode, respectively. The PW and WO electrodes can synchronously be colored rapidly to PB and WO\(_{\text{red}}\) resulting in the fluorescence intensity returning to the off state (Figure S2). The fluorescence intensity of the proposed FSDECEs can switch between the on and off states. The reversibly switching on and off states of...
FSDECEs can be achieved only by repeatedly connecting and disconnecting wires between the electrodes.

**RESULTS AND DISCUSSIONS**

Fluorescence switching based on double electrochromic electrodes (cathode and anode) (FSDECEs) was designed as follows. PB and WO electrodes were selected as the anode and cathode of the assembled FSDECEs, respectively. First, the electrochromic materials of PB and WO were electrodeposited on the fluorine-doped tin oxide (FTO) glass to obtain FTO/PB and FTO/WO electrodes (Scheme 1 and Scheme S1). The morphology of PB and WO films is observed in Figure 1a,d, which showed a smooth surface with several microcracks. The mappings of PB and WO electrodes can be seen in Figures S3 and S4. The corresponding mapping images of Fe, N, C, W, Mo, and O elements were consistent with lateral SEM images of PB (Figure 1b) and WO (Figure 1e). Considering that PB and WO matched with respect to synchronously bleaching and coloring, the time under the current density of electrophilic deposition of PB and the WO film was determined. And the thicknesses of the films were estimated to be approximately 440 nm (PB) and 463 nm (WO) in Figure S5. Notably, for the mapping of the W element, the high contrast is due to the signals of the Si element (existing FTO) being similar to those of the W element. XPS spectra were further used to analyze the components of the prepared electrochromic electrodes (Figure S6 and S7). XPS spectra of Fe 2p in the PB film are shown in Figure 1c. According to the previous reports, the peaks at
708.5 and 712.4 eV are ascribed to 2p_{3/2} of Fe^{3+} and Fe^{2+}. And the peaks at 724.9 and 721.3 eV are owing to the 2p_{1/2} of Fe^{3+} and Fe^{2+}. The XPS spectra of W 4f in the WO film appear at 37.7 and 35.5 eV due to W 4f_{5/2} and W 4f_{7/2} in Figure 1f. The above characterizations indicate that the two electrochromic electrodes are successfully prepared, and the structures of PB and WO materials make possible the insertion and extraction of ions and electrons.

The electrochromic process exhibits changes in the color and absorbance of the electrochromic material under proper potential stimulation, and the redox reactions take place with the insertion and extraction of ions and electrons. The schematic diagram for the coloring and bleaching processes of PB and WO electrodes is shown in Figure 2a. To further ascertain the feasibility of FSDECEs, the electrochromic property and cycling stability of the FTO/PB and FTO/WO single electrode should be investigated, respectively. The FTO/PB electrode possesses an obvious absorption in the range of 500−900 nm in the oxidation state (at 1.0 V) and almost no absorption in the reduced state (at −0.4 V) in Figure S8. Inversely, the FTO/WO electrode has an evident absorption over the range of 600−900 nm in the reduced state (at −1.0 V) but little absorption in the oxidized state (at 0.2 V) in Figure S9. The isochronous variation of the electrode color makes absorbance more distinct between coloring and bleaching states, and it can not only contribute to storing and releasing more energy when needed but also to enhancing the contrast of UV absorption in the coloring and bleaching states, which consequently provides a necessary prerequisite for the design of FSDECEs. These results confirmed the feasibility of assembling double electrochromic electrodes in a cell. Next, FTO/PB and FTO/WO electrodes are put together to assemble a double electrochromic electrode system, and the UV absorption spectra of FTO/PB | WO/FTO are shown in Figure 2c. The light-blue line indicates the absorbance of FTO/WO in the bleaching state and FTO/PB in the coloring state. The dark-blue line (in the coloring state) and gray line (in the bleaching state) represent FTO/PB and FTO/WO in the same state. It suggests that the double electrochromic electrodes possess a high-UV-absorption optical contrast in the coloring and bleaching states. By integrating the coloring electrodes of PB and WO as in the original state, the absorbance variation under double potential steps 0.5 V and −0.5 V (Figure 2d) is

\[
\text{Fe}_{3+}^{3\text{III}}\text{Fe}_{2+}^{2\text{II}}\text{(CN)}_6\text{Li}_4\text{(PB)} + 4\text{Li}^+ + 4e^- \rightarrow \text{Li}_4\text{Fe}_{3+}^{3\text{III}}\text{Fe}_{2+}^{2\text{II}}\text{(CN)}_6\text{Li}_4\text{(PW)}
\]

\[
\text{Li}_{a}\text{WO}_{3-a}\text{(WO}_{\text{red}}) \leftrightarrow \text{WO} + a\text{Li}^+ + a e^-
\]
performed. The contrast under coloring and bleaching states remains almost unchanged after 3600 s, showing the wonderful cycling stability and high optical modulation of the electrodes. Such excellent performance offers a guarantee for practical applications. The first and last cycles of Figure 2d are amplified in Figure 2e, and a very short response time is displayed, which is attributed to the synergistic effect of PB and WO in speeding up the electrochromic process to a degree.

After the synchronous bleaching process was achieved simply by connecting only the FTO/PB and FTO/WO red electrodes (Figure 3a), rapidly coloring after bleaching is a difficult challenge. Instead of a traditional approach such as adding oxidizing agents to accelerate the rate of coloring, here we use the Mg–O2 battery to charge the double electrochromic electrodes after discharge. This method could reduce the complexity of the system and eliminate the contrary effect of oxidizing agents for PB and WO electrodes. Besides, it is portable and durable, so the designed system can be realized via in situ application. To improve the performance of the Mg–O2 battery, it is urgent to employ a very efficient ORR catalyst. The FeN5 single atomic catalyst (SAC) was used.38 An SEM image of FeN5 SAC is shown in Figure 3b, and it catalyzes ORR at a potential of 0.6 V (vs RHE) (Figure 3c), which is efficient and comparable to that of commercial Pt/C but much cheaper and more accessible. The electron-transfer number (n) and selectivity of the catalysts toward H2O2 formation can be calculated according to the reaction occurring at the rotating-ring disk electrode:

\[
n = \frac{I_{\text{disk}}}{I_{\text{ring}} + 4N} \frac{I_{\text{ring}}}{N}
\]

\[
H_2O_2\% = 200 \frac{I_{\text{ring}}}{N^2I_{\text{disk}} + I_{\text{ring}}}
\]

On the basis of the experimental data in Figure 3c, we obtained \( n = 3.88 \) at 6.07% \( H_2O_2 \) (Figure S14). The calculation proves that \( n \) is close to 4, indicating \( O_2 \) through 4e\(^{-}\) transfer reduction, and directly produces \( H_2O \). The \( H_2O_2 \) % is very low and further clarifies that the \( O_2 \) can be reduced to \( H_2O \) in the Mg–O2 battery and the FeN5 single atomic catalyst has high-efficiency ORR activity. Besides, the choice of electrolyte solution is particularly important in shortening the response time of the system. After optimization, 1 M LiClO4 in propylene carbonate (PC) electrolyte solution containing 5% v/v \( H_2O \) is finally determined by taking the stability of the proposed system, the speed of the electrochromic process, and the occurrence of ORR into account. A small radius of the Li\(^+\) ion is suitable for quick insertion into and exit from the PB and the WO structure. This not only provides a proper electrolyte environment but also overcomes the formidable problem of the slow oxidation rate of PW to PB.35,36 In the optimized electrolyte, the power density of the Mg–O2 battery is high, up to \( 1.34 \) mW cm\(^{-2}\) (Figure 3e), and it can light an LED (Figure 3d). The large power density is sufficient to support the FTO/PB/WO/FTO electrodes with a power density of only \( 31.2 \) μW cm\(^{-2}\) (Figure 3f). After introducing the Mg–O2 battery.
with the FeN₅ SAC catalyst, the coloring and bleaching photographs of the double electrochromic electrodes are shown in Figure 3g. The obvious color change from blue to transparent can be observed by the naked eye. The charging process just need to connect the two wires of the Mg and WO electrodes as well as the PW and FeN₅ electrodes, while the discharging process occurs through connecting the PB and WO₆ electrodes. The in situ UV absorbance variation with time of the double electrochromic electrodes is exhibited in Figure 3h. After 30 cycles, the contrast can remain constant, showing good cycling stability. At the same time, the time of alteration between bleaching and coloring states is fast (Figure S15). The unexceptionable reversibility furnishes an essential foundation for the following fabrication of FSDECEs.

Next, fluorescent Ru@SiO₂ NP molecules are employed in the double electrochromic electrodes to assemble FSDECEs. The reason for choosing Ru@SiO₂ NPs as a fluorescent molecules is that its emission spectrum has a good coupling effect with the absorption spectra of electrochromic electrodes of PB and WO (Figure 4f). The prepared Ru@SiO₂ NPs have a uniform morphology, and the grain size is approximately 50 nm (Figure 4a and Figure S16). The HRTEM image and the EDS mappings of corresponding elements of Ru@SiO₂ NPs confirmed the successful preparation of Ru@SiO₂ NPs (Figure 4b). The fluorescence excitation spectra of Ru@SiO₂ NPs (black line) and the emission spectra (red line) are shown in Figure 4c. The fluorescence emission peak of Ru@SiO₂ NPs appears at 610 nm. The appropriate spectral overlap (Figure 4f) of donor and acceptors provides an indispensable condition for the regulation of interface fluorescence intensity. Layer-by-layer self-assembly is utilized to assemble the Ru@SiO₂ NPs on the double electrochromic electrodes to form FTO/PB/Ru@SiO₂ and FTO/WO/Ru@SiO₂ electrodes. Schematic diagrams of the assembly process and the related SEM image are shown in Figure 4d,e and Figure S17. The results suggest that the distance between Ru@SiO₂ NPs and the two electrochromic electrodes is as close as possible, which ensures that FRET occurs efficiently. Initially, when the PB and WO₆ electrodes are in the colored state, FRET occurs between fluorescent molecules and the electrochromic electrodes, resulting in a distinct decrease in the system fluorescence intensity. It is an off state in fluorescence switching. After FTO/PB/Ru@SiO₂ and FTO/WO₆/Ru@SiO₂ electrodes are connected, the PB and WO₆ electrodes are synchronously bleached and the absorbance becomes very low. Thus, no FRET occurs. The fluorescence of FSDECEs becomes much larger (on). The photographs of the two electrodes in the on and off states under a 360 nm UV lamp are exhibited in Figure 4g. Switching between two states can be accomplished by simply connecting and disconnecting the electrodes repeatedly. The fluorescence intensity variation of the FSDECEs with time is displayed in Figure 4h. The fluorescence contrast remains at 80% in the 30 cycles of tests, and the response times of off and on are 11.2 and 9.8 s, respectively (Figure S18). Also, a longer cycling stability of the FSDECEs (Figure S19) shows that the fluorescence contrast of the system is reduced slightly over 60 cycles of testing. The cycling stability is acceptable and is comparable to that of most single electrochromic electrode systems, which further exhibited the FSDECEs possessing long-term cycling stability and excellent fatigue resistance. Such superior performance can be attributed to the potential matching of PB and WO, the choice of electrolyte solution, the use of the FeN₅ single atomic catalyst, and the suitable spectral overlap of the fluorescent molecule and electrochromic materials.

■ CONCLUSIONS
Lithium-ion-assisted ultrafast charging double electrode smart windows with energy storage and a fluorescence display device (FSDECEs) are successfully fabricated for the first time. The FSDECEs (FTO/PB/Ru@SiO₂∥Ru@SiO₂/WO/FTO) demonstrate a high fluorescence contrast, short response time, and long-term cycling reversibility. Double electrochromic electrodes can store and release more energy when needed compared to single-electrode fluorescence switching. Synchronous bleaching and coloring of the two electrochromic electrodes exhibits a larger optical modulation, which allows the assembly of novel fluorescence switching based on double electrochromic electrodes. The fluorescence switching combining electrochemistry with fluorescence spectra has advantages of high sensitivity, ease of operation, and more portable in situ field applications. Such ingenious design and superior performance shed light on the next generation of transparent, visual, and portable devices.

■ ASSOCIATED CONTENT
* Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acscentsci.0c01149.

Materials and methods; additional figures including SEM images, TEM images, XPS characterization, assembly diagram of the electrodes, and schematic diagram of the working principle of the proposed system (PDF)

Self-bleaching/discharge and coloring/charge processes based on Mg−O₂ battery of the double electrochromic electrodes (PB and WO) were shown (AVI)

Coloring and bleaching processes of the PB and WO electrodes in the movie 1 were fast-forward in SI movie 2 (AVI)

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Notes
The authors declare no competing financial interest.

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