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Influences on oxidation voltage and holding time on poly(3-methylthiophene) film for electrochromic stability*

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In this study, we report the influences of oxidation potential and holding time on the electrochromic (EC) stability of poly(3-methylthiophene) (P3MT) film during the electrochemical reaction. The cycle stability and transmittance changes of the film were investigated by optimizing the oxidation potential, and its chemical compositions were measured by x-ray photoelectron spectra after multiple electrochemical cycles. High oxidation potentials can increase the P3MT film color contrast and decrease its cycle stability because of accelerating chemical decomposition. Moreover, the holding time with potential pulsing was analyzed by using the optical memory of P3MT at an optimized oxidation potential, which revealed the reduced voltage duration saved energy consumption by 11.6% and improved the EC cycle stability without changing in color contrast.

Keywords: electrochromism, P3MT, oxidation potential, cycle stability

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1. Introduction

Electrochromic (EC) material refers to that its optical property can reversible change in response to electrochemical reaction.[1,2] It is different from other passive color changing materials such as photochromic,[3,4] thermochromic,[5,6] and piezochromic ones,[7,8] because it can actively adjust the intensity of color depending on the use environment. Moreover, compared with other photoelectric materials, EC materials can be controlled by using a smaller voltage and keep the color for a period of time under open circuit condition, showing the memory effect.[9–14] Because of the above advantages, EC materials enable various useful applications such as rear-view mirrors,[15,16] information displays,[17,18] smart windows,[19–21] and military camouflage.[22,23]

In the midst of EC materials, conducting polymers such as polyaniline,[24] polycarbazole,[25] and polythiophene[26] have attracted more and more attentions due to their lots of advantages, which including multicolor capability and fast response time. However, the poor cycle stability of the EC conducting polymers during application limits the development of new EC materials, it is necessary to find a way to improve EC cycle stability. For instance, the cycle stability of EC conducting polymers can be improved by embedding transition metal elements such as Co^{2+}, Ni^{2+}, and Fe^{2+}.[27–30] Furthermore, the adhesive force of EC conducting polymers to working electrodes (fluorine doped tin oxide (FTO), indium tin oxide (ITO)) can be enhanced by chemical bonding to each other, leading to good EC stability.[21–34] Combination of EC conducting polymers and carbon nanomaterials (graphene oxide and functionalized graphene) have exhibited good long-term optical stability,[35,36] which can be better improved by selecting a right electrolyte.[37–42]

The electrolyte and electrode modification can enhance the operating voltages control efficiency to achieve satisfactory stability in EC process, with inappropriate voltage often causing irreversible EC reaction and undermining the EC material stability.[43] However, although many valuable studies have been explored about the above stability issues, the regulation of driving voltage has not been investigated in detail. In this paper, the influences of oxidation voltages and holding time on EC electrochemical reaction were discussed for poly(3-methylthiophene) (P3MT) films, and the long-term cycle stability of P3MT films was attained by analyzing the relevant parameters. Moreover, the good optical memory of P3MT was closely related to voltage holding time in EC reaction, which reduced the energy loss by 11.6%.

2. Experimental procedures

All solvents and chemicals were of analytical grade and used without further purification. P3MT was purchased from Sinopharm Chemical Reagent Co., Ltd. ITO-coated glass (20 mm×40 mm, 15 Ω/sq, China) was successively washed with acetone, ethanol, and de-ionized water in an ultrasonic bath for 15 min. The cleaned ITO substrate was spin coated by spin coating instrument (KW-4C, SETCAS Electronics Co., Ltd.) with a 0.8-wt% P3MT chlorobenzene solution

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(1200 rpm, 20 s) and dried in an oven at 70 °C for 15 min. The thickness of P3MT film about 50 nm was characterized by thickness gauge (CHY-U, Sumspring Co., Ltd.). Electrochemical properties were measured by electrochemical cells using the electrolyte 1M lithium perchlorate ($LiClO_4$) dissolved in propylene carbonate (PC), with P3MT on ITO glass as the working electrode, Ag/AgCl electrode as the reference electrode, and Pt foil as the counter-electrode, respectively. Cyclic voltammetry (CV) curves were acquired at different voltages at a scan rate of 40 $mV{\cdot}s^{-1}$. EC characterization was measured via a UV-Vis spectrophotometer (2450, Shimadzu) analyzing the transmittance at a wavelength of 590 nm. The chemical compositions of sample was carried out using x-ray photoelectron spectrometer (XPS, PHI-5700) with a dual Mg–Kα–Al–Kα anode for photo-excitation.

3. Results and discussion

The CV curves of P3MT films prepared under different voltage conditions are shown in Fig. 1(a), with the minimum voltage set at $E_0 = -0.3$ V, and the maximum voltage (E_1) changed from 0.5 V to 0.9 V. It can be seen that the reduction peaks at 0 V and 0.4 V and oxidation peaks at 0.1 V and 0.5 V in the electrochemical reactions of P3MT, indicating the transition state of P3MT polarons and bipolarons.[29] Moreover, the color of the P3MT film was changed from green to blue gradually when applied voltages from E_0 to E_1, and the color regained its original state when applied reverse voltages from E_1 to E_0. Therefore, these results suggested that P3MT was involved in the following chemical reaction under varying voltage conditions:[29,30]

$$\mathrm{P3MT^0} + \mathrm{ClO_4^-} \underset{\text{reduction}}{\overset{\text{oxidation}}{\rightleftharpoons}} \mathrm{P3MT^+ClO_4^-\ (polaron)} \underset{\text{reduction}}{\overset{\text{oxidation}}{\rightleftharpoons}} \mathrm{P3MT^{2+}2ClO_4^-\ (bipolaron)}$$

To determine optimal E_1 value for the EC reaction of P3MT film, figure 1(b) shows transmittance (T) at 590 nm measured with voltage (E_1) varied between 0.5 V and 0.9 V, indicating that a positive correlation between T from 73.57% to 85.77% (transmittance change $\Delta T = 12.20\%$) and voltages from 0.1 V to 0.5 V. Furthermore, the greater the voltage, the greater the corresponding oxidation current density. However, more than 0.5 V, the T increased slowly and only slight changes observed from 0.7 V ($T = 87.21\%$) to 0.9 V ($T = 88.70\%$). From Fig. 1(a) and Fig. 1(b), it can be concluded that the color of P3MT film is rapidly changed before reaching the highest current density and the EC reactions gradually reach saturation. According to the experimental result of potential pulse with time interval of 25 s from E_0 to E_1, we can clearly understand the relationship between the ΔT and the E_1 (Fig. 1(c)). At $E_1 = 0.5$ V, the P3MT film has a ΔT of 23.7% for bleached/colored states (Table 1). At $E_1 = 0.7$ V, the bleached state T increases significantly, leading to $\Delta T = 25.9\%$. While at $E_1 = 0.8$ V–0.9 V, ΔT is almost constant (26.4%–26.7%). These findings indicate that the EC reaction reaches saturation when E_1 is greater than 0.7 V, which is consistent with the observed optical changes in the cyclic voltammetry experiment (Fig. 1(d)).

The optimization of oxidation potential is of great importance to P3MT film EC lifetime, which was investigated by detecting multiple cycle stability more than potential pulsing 400 cycles (Fig. 2). Record the initial states ΔT values and that after 200 cycles at varying E_1 with per cycle interval of 50 s (25 s for colored and bleached states). It can be seen that the initial ΔT and that after 200 cycles are 26.2% and 24.7% when E_1 is 0.7 V, which leads to $\Delta\Delta T$(difference in ΔT) = 1.5% (Fig. 2(a)). When E_1 is 0.8 V, the initial ΔT is 26.9%, decreasing to 21.3% after 200 cycles, and the corresponding $\Delta\Delta T$ is 5.6% (Fig. 2(b)). However, for E_1 is 0.9 V, $\Delta\Delta T$ obviously increases to 19.4% (from 27.3% to 7.9%) (Fig. 2(c)). With E_1 changes from 0.7 V to 0.9 V, the initial ΔT has a slight increase (1.1%), but there is a substantial increase (16.8%) in that after 200 cycles. Therefore, from the above analysis, it can be deduced that the increase of E_1 leads to poor P3MT film cycle stability in certain circumstances.

Table 1. Electrochromic and electrochemical parameters for P3MT film at varying E_1. t_c and t_b denote the coloration time and the bleaching time, respectively, and the switching time is defined as the time required for 90% of the total transmittance.

E_1 / V	ΔT/%	t_c/s	t_b/s	Q_a/mC·cm^{-2}	Q_a/mC·cm^{-2}	Q_c/Q_a
0.5	23.7	0.49	0.89	0.318	0.321	0.992
0.7	25.9	0.66	0.77	0.497	0.507	0.981
0.8	26.4	0.67	0.78	0.551	0.568	0.970
0.9	26.7	0.69	0.76	0.632	0.658	0.961

It is well known that the electrochemical reversible stability, related to the ion storage capacity of the film, is a good indicator of the electrochemical activity of the electrode, which is expressed as the ratio between the cathodic and anodic charge densities (Q_c/Q_a). As E_1 increased from 0.5 V to 0.9 V, the Q_c/Q_a decreased (Table 1), which could cause irreversible reaction when the oxidation potentials increased over 0.5 V (the highest current density). To further study the irreversible reaction, the chemical compositions of films were conducted by x-ray photoelectron spectra (Fig. 3). The red lines represent the original reference peaks of the film. The XPS spectra for the film under different voltages were fitted by Origin 8.5 with

Gaussian-Lorentzian function and referenced to C 1s peak located at 284.8 eV. The as-prepared P3MT film shows S $2p_{3/2}$ and $2p_{1/2}$ peaks at 163.78 eV and 165.17 (Fig. 3(a)), respectively, which are the characteristic peaks of thiophene.[31–33] After 450 cycles at $E_1 = 0.7$ V (Fig. 3(b)), two new peaks appeared near 165.01 eV (S $2p_{3/2}$) and 166.13 eV (S $2p_{1/2}$), which are ascribed to sulfinyl (R_2–SO), is due to the oxidation of thiophene to sulfinyl (R_2–SO). This can be considered as a transient state from thiophene to sulfone (C–SO_2–C), a catabolite of P3MT.[31–34] The film with the same number of cycles at $E_1 = 0.8$ V exhibited new C–SO_2–C peaks at 167.96 eV (S $2p_{3/2}$) and 169.31 eV (S $2p_{1/2}$) (Fig. 3(c)). These peak intensities were greatly increased when P3MT be further oxidized at 0.9 V (Fig. 3(d)), which implied it accelerates chemical decomposition. Furthermore, sulfide (C–S–C) peaks appeared at 163.90 eV (S $2p_{3/2}$) and 164.93 eV (S $2p_{1/2}$), with the significantly weakened thiophene peaks indicating that the P3MT molecular chain was completely destroyed by electrochemical oxidation.[44–47] These results indicate that EC reactions caused the P3MT chemical decomposition, and the higher the oxidation potential, the faster the decomposition.

Table 2 shows the area ratios of sulfur groups, which be calculated from the S 2p XPS spectra of the film under different voltages. Their relative areas can be correlated with the valence of S element. It is found that the vibration peak of C_4S–H decreases while the peaks of C–SO_2–C and C–S–C increase with the potential increases, which is in agreement with the results of the above analyses.

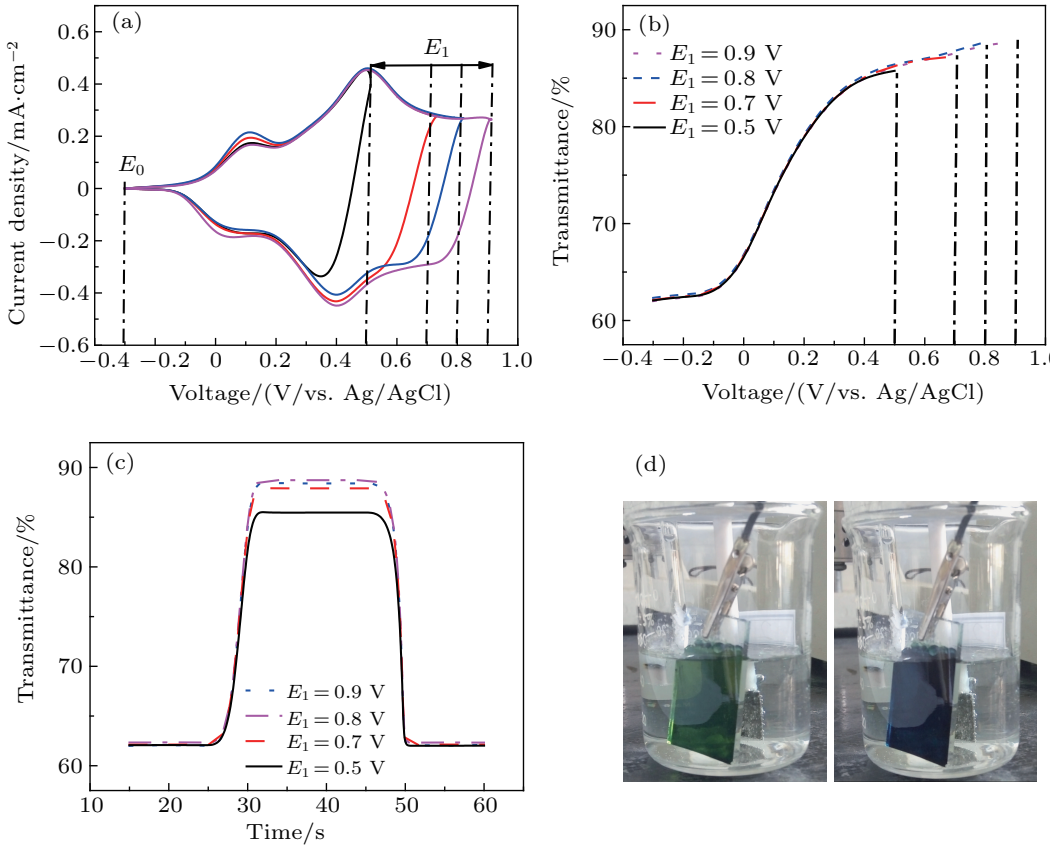


Fig. 1. (color online) (a) *CV* curves of P3MT films measured from E_0 (−0.3 V) to E_1 (0.5, 0.7, 0.8, and 0.9 V). (b) Transmittance changes during *CV* measurement and (c) the result of potential pulse test. (d) Optical images of colored (blue) and bleached (green) states for P3MT film on ITO.

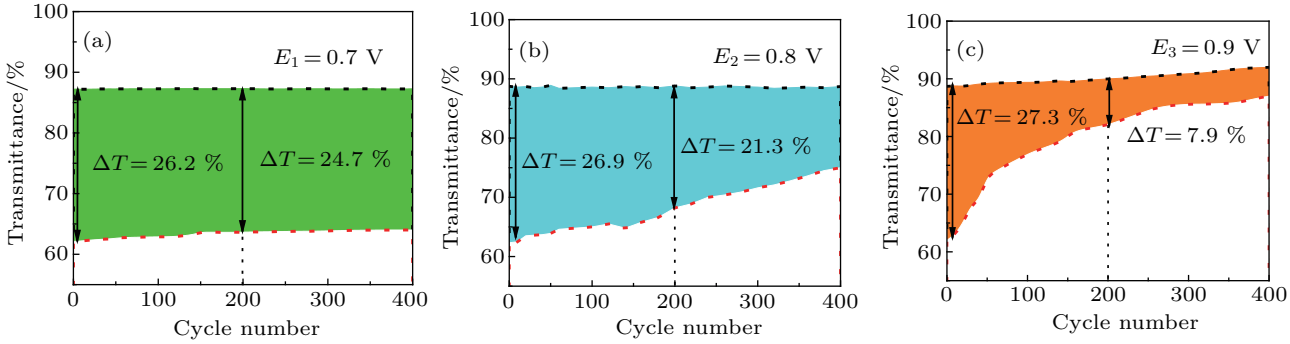


Fig. 2. (color online) Long-term cycle stability of P3MT films at $E_1 = 0.7$ (a), 0.8 V (b), and 0.9 V (c).

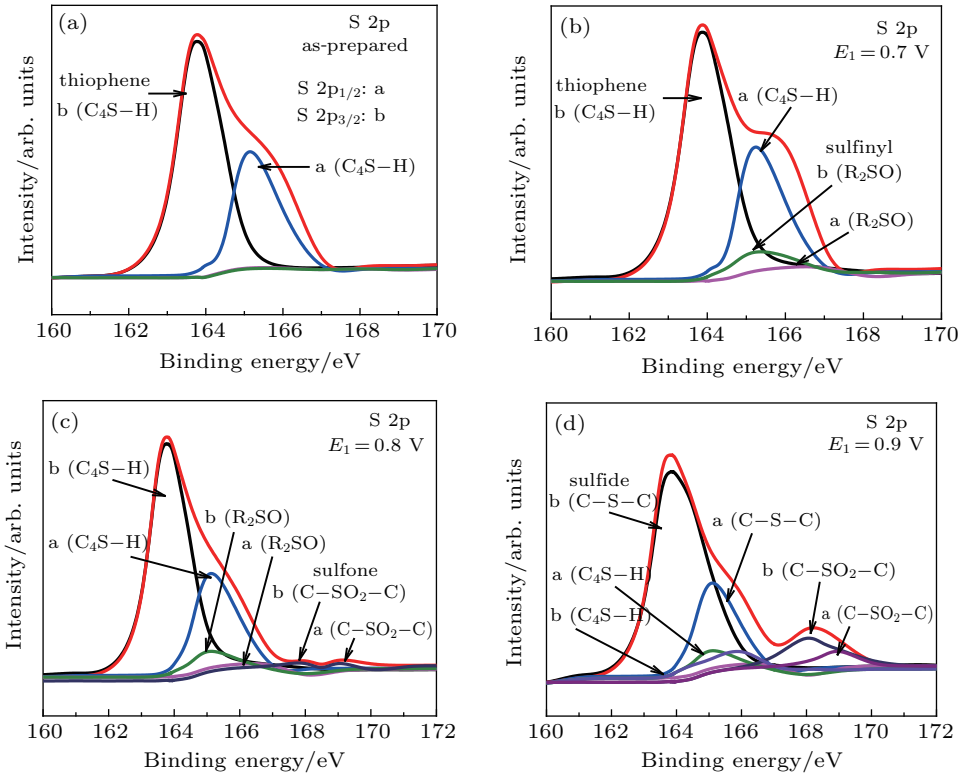


Fig. 3. (color online) S 2p XPS spectra of (a) as-prepared P3MT film and P3MT films after 450 cycles between −0.3 V and $E_1 = 0.7$ V (b), 0.8 V (c), and 0.9 V (d).

Table 2. The area ratio of each peak in the S 2p XPS spectra under different voltages.

Voltage/V	Area ratio/%							
	C_4S–H (S^{2+})		R_2SO (S^{4+})		C–SO_2–C (S^{6+})		C–S–C (S^{2-})	
	163.78/eV	165.17/eV	165.01/eV	166.13/eV	167.96/eV	169.31/eV	163.9/eV	164.93/eV
–0.3	65.21	34.79	–	–	–	–	–	–
0.7	60.34	25.79	10.36	3.51	–	–	–	–
0.8	57.06	23.09	11.65	4.47	2.12	1.61	–	–
0.9	2.64	0.89	–	–	7.74	2.92	60.09	25.72

Expected the holding time also influences the cycle stability of P3MT at the optimized voltage is reasonable. The EC film still maintains its color when disconnected the voltage, *i.e.*, exhibits the so-called optical memory, which allows to control the EC reaction holding time. Figure 4(a) shows the optical transmittance of P3MT after applying a voltage of 0.7 V for 25 s. The transmittance of P3MT remains at 87.22% when voltage continues to be applied. Moreover, the bleached state transmittance has only changed by 0.4% at the sixth minute when the voltage is disconnected. Thus, it is concluded that P3MT film possesses an excellent optical memory.

Figure 4(b) presents the effects of voltage holding time with varying voltages. In the experiment, film T_1 is studied using pulse potential steps under continuous voltage, but the films T_2, T_3, and T_4 disconnect voltage in the tenth second at −0.3 V and are studied by changing oxidation voltage durations (25, 10, and 5 s) at 0.7 V. The optical transmittance of these films under varying potential pulsing conditions is shown in Fig. 4(c). It implies that there is no notable difference in ΔT and the voltage holding time has no influence on EC coloring/fading in the process of initial pulse potential. Interestingly, voltage holding time has a notable effect on long-term cycle stability (Figs. 4(d) and 4(e)). For film T_1, ΔT after 1000 cycles (ΔT_b) is 15.6%, and the ratio of ΔT_b to initial ΔT_a is 0.59. However, in the T_2, T_3, and T_4 cases, ΔT_b are 18.1%, 20.2%, and 23.5%, with $\Delta T_b/\Delta T_a$ equals 0.67, 0.73, and 0.88, respectively. In fact, Excessive holding time tends to inject too many ions and electrons into the film, so that ions and electrons cannot be completely extracted from the film and they accumulate in the film to block the diffusion channels, causing the electrochemical performance and cycle stability of the film to decrease. Meanwhile, the electrochromic process is a reversible electrochemical reaction process. The microstructure, optical properties and conductivity of the film change when the voltage is repeatedly applied, and the reduction of the holding time is equivalent to reducing the effect of the ap-

plied voltage on the film and increasing its long-term cycle stability. Besides, the excessive holding time leads to the conductive glass (ITO) as an electrode to undergo electrochemical reaction and corrosion of the electrolyte solution, causing the path of electron transport to be destroyed and a decrease cycle stability for the film. These imply that the decrease of voltage holding time leads to the improvement of P3MT cycle stability. Additionally, the film with the shortest holding time (T_4) can save energy consumption by 11.6% (Table 3). Thus, the excellent optical memory of P3MT film can be compatible with the high cycle stability and low power consumption of the film.

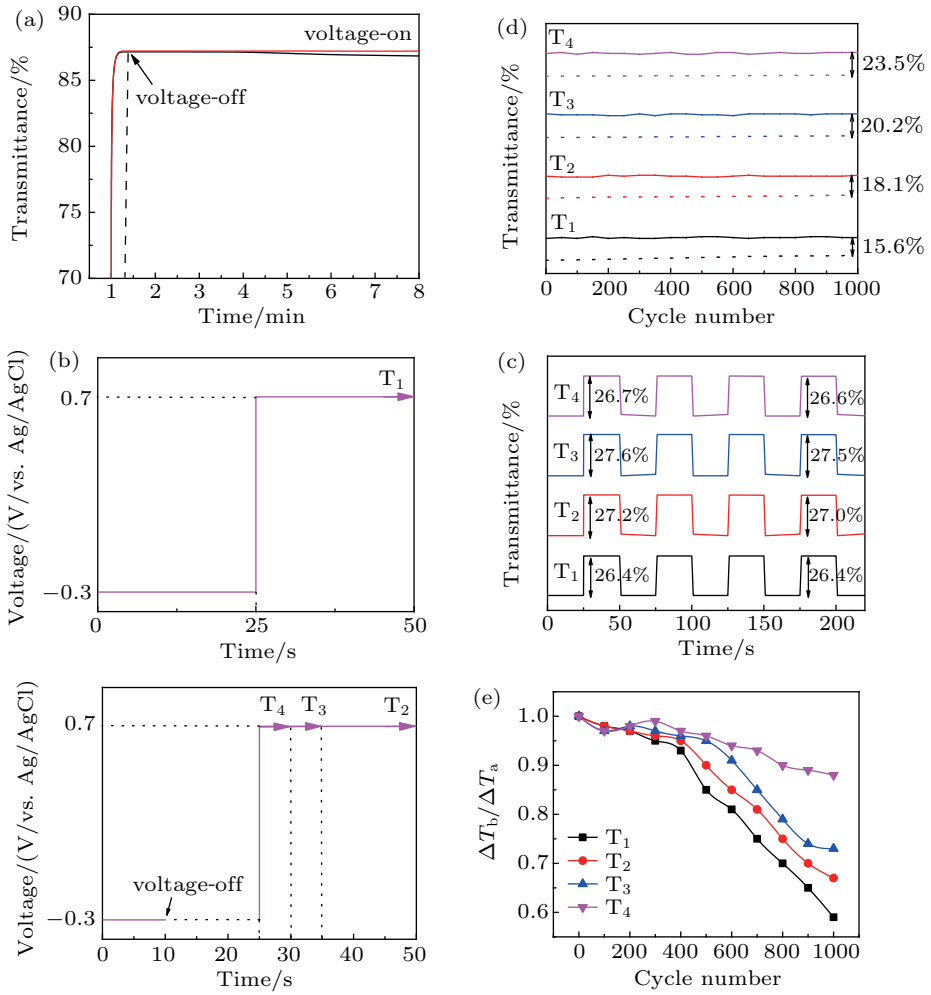


Fig. 4. (color online) (a) Transmittance difference of P3MT films at voltages on and off, (b) potential pulse at different voltage holding times (T_1–T_4) and the corresponding transmittance difference for films undergone (c) four initial cycles and (d) long-term cycles (solid curves, bleached state; dashed curves, colored state). (e) Standardized transmittance difference as a function of cycle number.

Table 3. Power consumption and reduced power consumption of different P3MT films.

	T_1	T_2	T_3	T_4
Power consumption/nWh·cm^{-2}	39.7	39.2	37.3	35.1
Reduced power consumption/%	0.0	1.2	6.1	11.6

4. Conclusions

In this work, we have studied the influences of voltage factors (oxidation potential and holding time) on the EC cycle stability of P3MT film, indicating that higher oxidation voltage leads to higher transmittance difference and worse lifetime. The decline of long-term cycle stability in excessive oxidation experiment is attributed to the decomposition of P3MT to produce sulfone and sulfide. In addition, the excellent optical memory of P3MT film allows the cycle stability of the film to be improved and the power consumption to be reduced. Thus, Optimizing oxidation potential and holding time is one of the most convenient ways to improve the long-term cycle stability of EC devices without any EC material and electrolyte modification.

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