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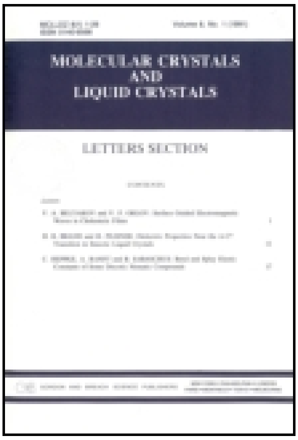

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Improved Open-Circuit Voltage of Dye-Sensitized Solar Cells with $LiNO_3$-Incorporated Titanium Dioxides

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Improved Open-Circuit Voltage of Dye-Sensitized Solar Cells with $LiNO_3$-Incorporated Titanium Dioxides

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The influence of $LiNO_3$ incorporation onto TiO_2 electrodes on the performance of dye-sensitized solar cells (DSSCs) was studied. $LiNO_3$-incorporated TiO_2 electrodes were prepared by a dip coating process and the resulting electrodes were applied to DSSCs. Open circuit voltage (V_{oc}) of DSSC with a $LiNO_3$-modified TiO_2 layer was averagely improved by over 24% compared to that of a reference device with bare TiO_2. The conduction band edge shift of TiO_2 to a negative direction, arising from the formation of a surface dipole, accounted for the improvement in V_{oc}.

Keywords Conduction band edge shift; dye-sensitized solar cell; lithium nitrate; open circuit voltage

Introduction

Dye-sensitized solar cells (DSSCs) based on nanocrystalline porous TiO_2 films are regarded as a potential low-cost alternative to conventional inorganic solar cells [1–6]. An impressive 12.3% solar-to-electric power conversion efficiency (PCE) has been reported [7]. However, further improvements in PCE are necessary to successfully commercialize DSSCs. TiO_2 photoanodes in these cells plays an important role on the photovoltaic performance. High porosity of the TiO_2 layer provides extended surface area for dye adsorption, resulting in enhancing light harvesting efficiency of DSSCs. TiO_2 is also a good transporter of electrons injected from the photoexcited dyes to TiO_2 conduction band. Thus, high short circuit current (J_{sc}) can be achieved from the TiO_2 cells. However, open circuit voltage (V_{oc}) in DSSCs with TiO_2 layer maintains low values. As a well-known method to improve V_{oc}, interfacial modifications to the TiO_2 layer have been conducted, resulting in the conduction band edge shift of the TiO_2 or suppression of charge recombination. There are three cases in

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which surface modifications can influence the V_{oc} value of DSSCs with a surface-modified TiO_2 layer. In the first case, materials such as $CaCO_3$ and Nb_2O_5 form an energy barrier [8,9]. The electrons injected into the TiO_2 layer may transfer back to the sensitizer or to the electrolyte due to an electron recombination phenomenon, i.e., $I_3^- + 2e^-_{cb}\,(TiO_2) \rightarrow 3I^-$. The energy barrier causes deceleration in the recombination rate for a given electron population, resulting in a larger concentration of electrons in the TiO_2 layer. This increase induces a more negative Fermi level in the TiO_2 and thus a larger V_{oc}. In the second case, the surface dipoles could be shaped instead of the energy barriers, depending on the modification process. Some oxide compounds, such as $SrTiO_3$ and ZnO, have been reported to make surface dipoles [10,11]. Surface dipoles have been shown to shift the conduction band edge of TiO_2 toward a negative potential. In this case, a back electron transfer is not hindered, but overall conversion efficiency is improved due to the enhancement of V_{oc} [12,13]. In the last case, surface-coating materials passivate the surface states, which are considered to be the recombination centers, leading to an enhancement of V_{oc} [14].

In this paper, we selected lithium nitrate ($LiNO_3$) as a surface modification material (surface modifier). The TiO_2 surfaces were directly modified via a very simple dip coating process using aqueous $LiNO_3$ solution, and the resulting electrodes were applied to the photoanodes of DSSCs. We expected the $LiNO_3$ on the electrodes to possibly behave as surface dipoles or as an insulation layer on the TiO_2 layers, resulting in an improvement in V_{oc} of the DSSCs. Thus, we examined whether $LiNO_3$ is successfully deposited onto the TiO_2 layers by simple dip coating, and the effects of the surface treatment on the performance of the DSSCs were also investigated.

Experimental Details

Materials

Commercial TiO_2 pastes [T20/SP (20 nm), Ti-nanoxide 300 (400 nm); Solaronix] and $LiNO_3$ (Sigma-Aldrich) were selected as photoelectrode and coating material, respectively. Commercial N719 dye [$RuL_2(NCS)_2(TBA)_2H_2{\cdot}4H_2O$, L = 2,2′-bipyridyl-4,4′-dicarboxylate, TBA = tetrabutylammonium; Solaronix] was employed as the sensitizer. Iodide-based commercial electrolyte (AN-50; Solaronix) and a Pt source ($H_2PtCl_6{\cdot}5.5H_2O$; KOJIMA CHEMICALS) were selected. All of the chemicals were used without any further purification.

Preparation of DSSCs

To prepare the working electrodes, fluorine-doped tin oxide (FTO, sheet resistance: ∼10 $\Omega/\square$) glasses were cleaned in a detergent solution using sonication for 20 min, and then thoroughly rinsed with deionized water (DI) and ethanol. After treatment with UV-O_3 for 20 min, the FTO glasses were immersed into a 40 mM $TiCl_4$ solution at 70 °C for 30 min and then washed with DI and ethanol. An active TiO_2 layer was formed on the FTO glass via a doctor-blade method followed by a calcining process at 500 °C for 60 min. An additional TiO_2 layer composed of ca. 400-nm particles was then deposited and subsequently calcinated. Finally, the TiO_2 films were treated with 40 mM $TiCl_4$ solution again and annealed at 500 °C for 60 min to prepare the TiO_2/FTO electrodes. The electrodes were soaked in aqueous solutions (0.05 M) of $LiNO_3$ for 1∼20 min to deposit the $LiNO_3$ onto the TiO_2 layers. The resulting electrodes were rinsed with DI and ethanol, and dried at 65 °C for

Table 1. Amount of $LiNO_3$ coated onto the TiO_2 layer

Applied electrodes	dipping time (min)	Amount of $LiNO_3$ (wt%)
TiO_2/FTO	0	0
$LiNO_3$-TiO_2/FTO		
$LiNO_3$(1)-TiO_2/FTO	1	0.86
$LiNO_3$(5)-TiO_2/FTO	5	0.93
$LiNO_3$(10)-TiO_2/FTO	10	0.96
$LiNO_3$(20)-TiO_2/FTO	20	1.13

10 min to prepare the $LiNO_3$-modified TiO_2 layer ($LiNO_3$-TiO_2/FTO). Both bare TiO_2/FTO and $LiNO_3$-TiO_2/FTO electrodes were separately immersed in a 0.5 mM N719 dye solution (acetonitrile/tert-butyl alcohol, v/v = 1/1) for 24 h. To prepare the counter electrode, two holes were drilled in the FTO glass and cleaned with the method described previously. A drop of 0.7 mM H_2PtCl_6/isopropanol solution was placed on the rinsed FTO glass, and then the glass was calcined at 400 °C for 30 min. Thermally treated platinum counter electrodes were placed on the TiO_2/FTO electrodes, and these were then sealed with a 60 μm-thick sealing material (SX1170–60PF; Solaronix). The electrolyte was introduced into the cells through one of the two small holes drilled in the counter electrodes to give DSSCs with 25 mm^2 active area.

Measurements

X-ray photoelectron spectroscopy (XPS) was performed using a VG Multilab ESCA 2000 (ThermoVG Scientific) with Mg Kα radiation. The C 1s photoelectron peak (binding energy at 284.6 eV) was used as an energy reference. The amount of $LiNO_3$ introduced onto the TiO_2 layer was measured using inductively coupled plasma mass spectrometry (ICP/MS, Elan 6100, PerkinElmer). To calculate an amount of absorbed dye molecules, UV-Vis diffuse reflectance spectra (UV-Vis DRS) were obtained from a Lambda 750 UV-Vis spectrophotometer (PerkinElmer) equipped with an internal diffuse reflectance integrating sphere. Photocurrent-voltage measurements were performed using a Keithley model 2400 Source Meter and a Newport 91192 solar simulator system equipped with a 1 kW xenon arc lamp (Oriel). Light intensity was adjusted to 1 sun (100 $mWcm^{-2}$) with a Radiant Power Energy Meter (model 70260, Oriel). The incident photo-to-current conversion efficiency (IPCE) results were acquired using an IPCE G1218a (PV Measurement). This system applies monochromatic light from a 75W xenon arc lamp (Ushio UXL-75XE), which is filtered by a dual-grafting monochromator and individual filters, onto the test devices. An ellipsoidal reflector collects light from the lamp and focuses it on the monochromatic entrance slit via a mechanical chopper to produce a small, modulated signal. As the modulated, monochromatic light was applied to the test devices, a continuous bias light (1 sun) was also applied.

Results and Discussion

TiO_2/FTO electrodes were soaked in aqueous solutions of $LiNO_3$, and the rinsed electrodes were dried at 65 °C to produce the $LiNO_3$-TiO_2/FTO. We prepared four different $LiNO_3$-TiO_2/FTO electrodes by varying the soaking time from 1 to 20 min. The amount of $LiNO_3$

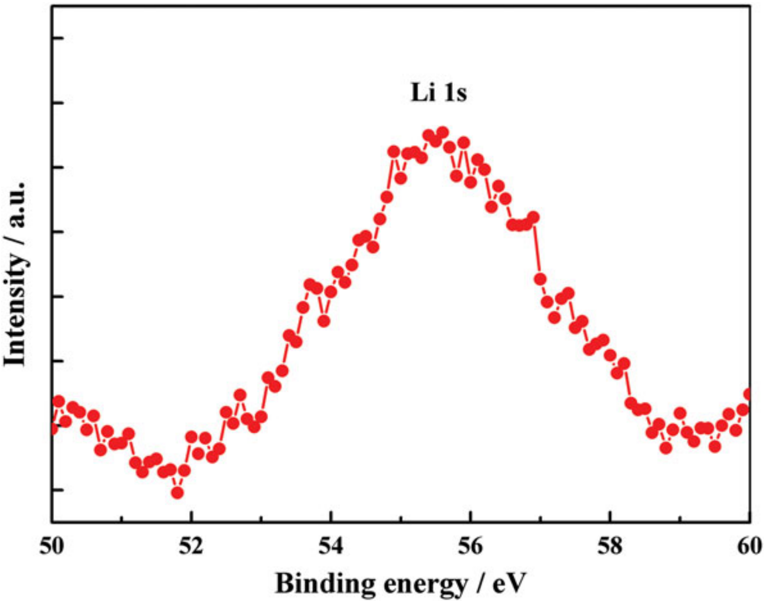

Figure 1. X-ray photoelectron spectrum for the Li 1s peak in $LiNO_3$-TiO_2/FTO.

incorporated onto the TiO_2 layer was calculated using the data from the ICP measurements, where the results confirmed that the amount of $LiNO_3$ was slightly increased by extending the soaking time (Table 1). XPS measurements of the $LiNO_3$-modified electrode were also conducted. As an evidence of $LiNO_3$ incorporation, the characterization peak for the Li 1s was clearly detected at 55.6 eV (Fig. 1).

The DSSCs were fabricated with $LiNO_3$-TiO_2/FTO and bare TiO_2/FTO electrodes, and their photovoltaic properties were characterized. The average photovoltaic properties of the DSSCs as a function of surface-modification time are presented in Fig. 2. As can be seen from Fig. 2(a), the V_{oc} values were increased by the treatment of TiO_2 surface with dilute $LiNO_3$ solution. The V_{oc} enhancement of about 24.6% on average was achieved in all the surface-treated devices, as compared to the reference cell with bare-TiO_2/FTO, i.e., without any treatment. However, the J_{sc} and fill factor (FF) values were observed to decrease, as shown in Fig. 2(b) and 2(c). The overall efficiencies of DSSCs with $LiNO_3$-TiO_2/FTO were enhanced from that of the reference cell, because the improvement in V_{oc} overrode the reduction in J_{sc} and FF. The DSSCs with $LiNO_3$(10)-TiO_2/FTO electrodes, corresponding to a soaking time of 10 min, showed the better performance [Fig. 2(d)], resulting in an improvement in the PCE (7.40%), compared to that of the reference device (6.88%). Therefore, we focused on the device with $LiNO_3$(10)-TiO_2/FTO to reveal the origin of an enhancement in the PCE. Figure 3 shows the current density (J) and voltage (V) curves of DSSCs with $LiNO_3$(10)-TiO_2/FTO and bare TiO_2/FTO electrodes, and the device performances are compared in Table 2.

As described previously, V_{oc} could be affected by the conduction band edge shift of the TiO_2 or suppression of charge recombination. Thus, we first investigated whether the $LiNO_3$ modification induced band edge shift or charge recombination. Electrochemical impedance spectroscopy (EIS) has been widely used for investigating the kinetics and energetics of transport and recombination in DSSCs [15,16]. Figure 4 shows the Bode

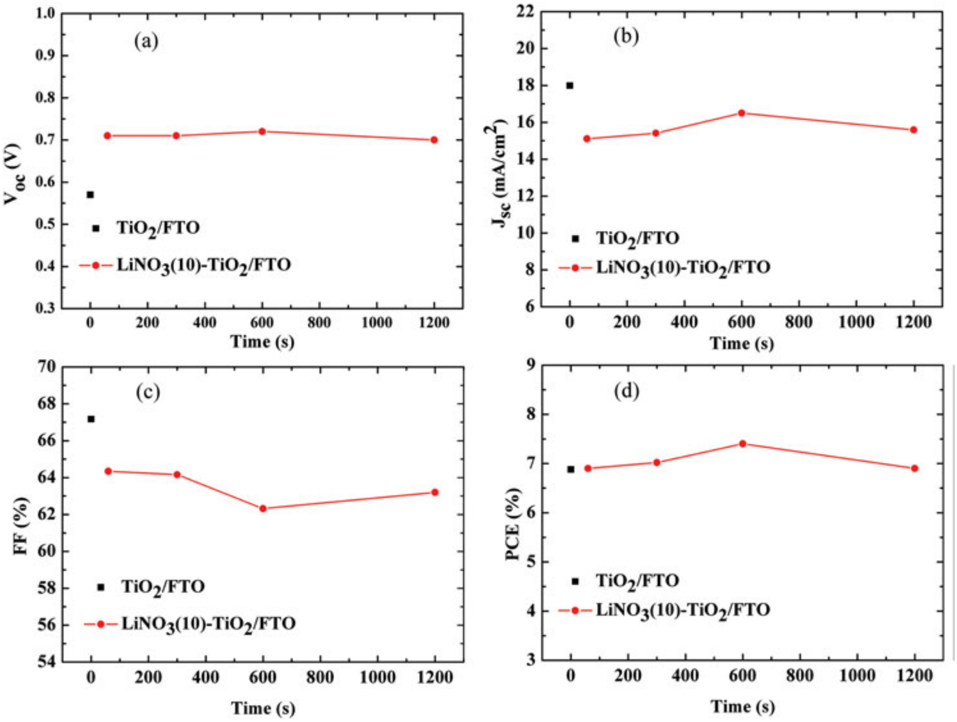

Figure 2. Performance variations with dipping time; (a) V_{oc}, (b) J_{sc}, (c) FF and (d) PCE of DSSCs characterized under AM 1.5 irradiation.

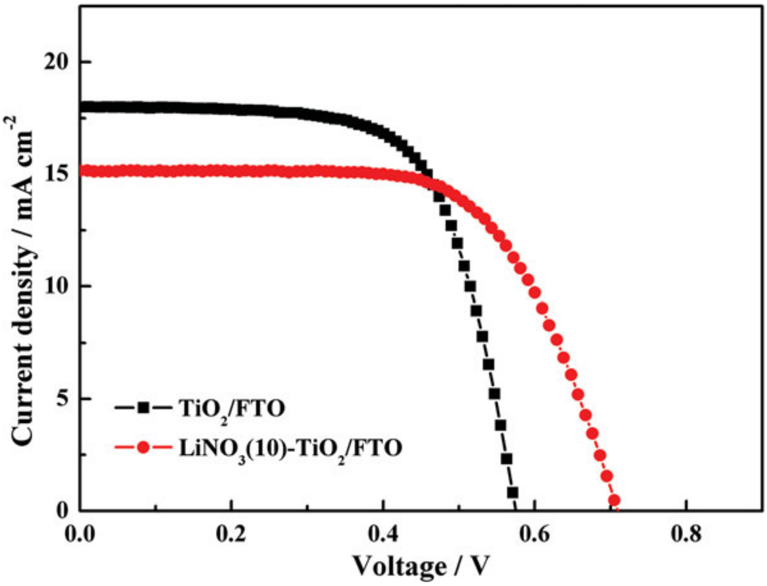

Figure 3. J-V characteristics of DSSCs with bare and $LiNO_3$-modified TiO_2 layers.

Table 2. Performance comparison of the DSSCs employing TiO_2/FTO and $LiNO_3$(10)-TiO_2/FTO as photoanodes

Applied electrodes	V_{oc} (V)	J_{sc} (mA cm^{-2})	*FF* (%)	η (%)	Loaded dye (mol cm^{-2})
TiO_2/FTO	0.57	17.99	67.17	6.88	1.82×10^{-7}
$LiNO_3$(10)-TiO_2/FTO	0.72	16.50	62.32	7.40	1.87×10^{-7}

phase plots of the EIS spectra (mid frequency range) in an open-circuit condition under the illumination of simulated AM 1.5 solar light (100 mA/cm^2). The frequency peak related to the $LiNO_3$(10)-modified TiO_2 electrode was almost equivalent to that of bare TiO_2/FTO. The electron lifetime (τ_n) was estimated from the equation $\tau_n = 1/(2\pi f_{max})$, where f_{max} is the peak frequency. The f_{max} values of the DSSCs with $LiNO_3$(10)-TiO_2/FTO and TiO_2/FTO were measured to be 13.101 and 13.102 Hz, and thus the electron lifetime was calculated to be 12.148 and 12.147 ms, respectively, which were almost identical. When we considered that the energy barrier causes an increment in the electron lifetime of the TiO_2 [17], we concluded that our $LiNO_3$ modification of the TiO_2 does not induce formation of an energy barrier.

Although the dark current of DSSCs does not directly reflect the recombination current under illumination, it can be used to measure a conduction band shift [10]. Figure 5 presents the dark current-voltage characteristics of the DSSCs with bare TiO_2/FTO and $LiNO_3$(10)-TiO_2/FTO as photoanodes. The onset potential of the dark current for the bare TiO_2/FTO electrode was measured to be ca. 0.45 V, whereas the dark current potential for the $LiNO_3$(10)-TiO_2/FTO electrode was shifted to ca. 0.53 V. The $LiNO_3$ modification induces a shift of the onset potential to a higher voltage, indicating that electron injection from FTO electrode to TiO_2 layer in DSSC with $LiNO_3$(10)-TiO_2/FTO is more unfavorable.

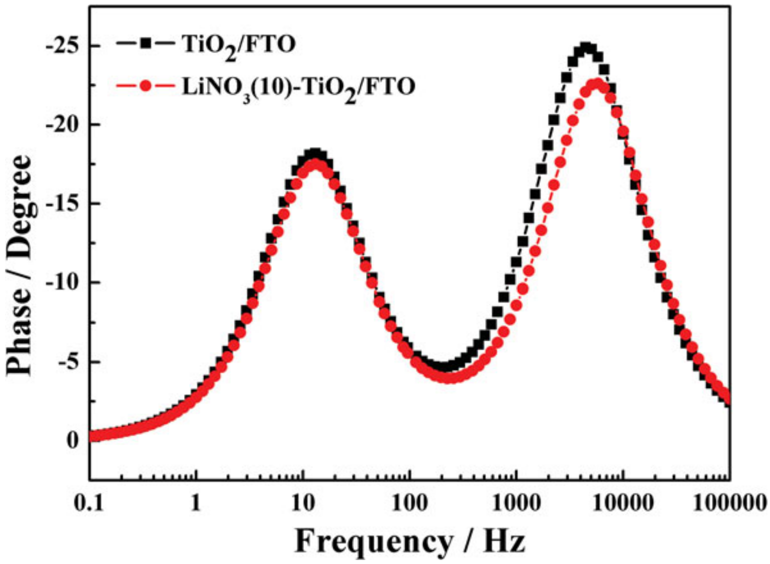

Figure 4. Bode plots of EIS spectra for DSSCs with bare TiO_2 and $LiNO_3$-modified TiO_2 electrodes.

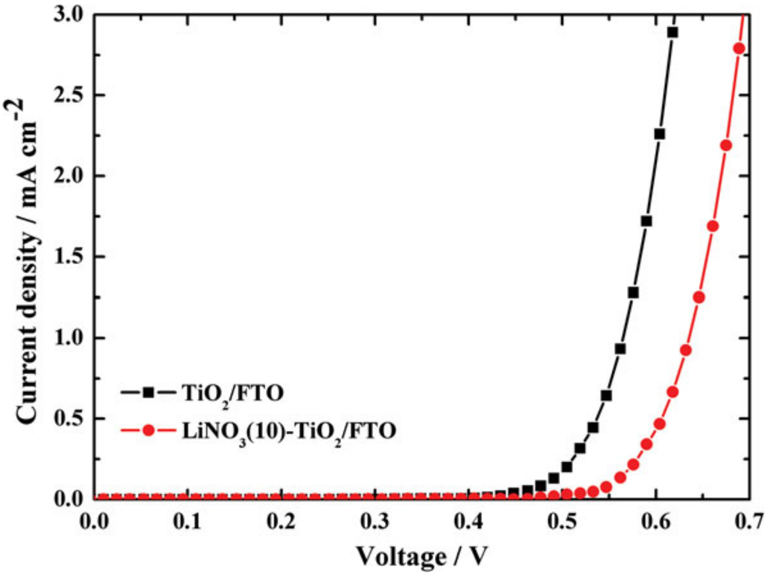

Figure 5. Dark current-voltage characteristics of DSSCs with bare and $LiNO_3$-modified TiO_2 layers.

This fact implies that the $LiNO_3$ modification shifts the conduction band edges of TiO_2 in the negative direction, resulting in the larger potential difference between TiO_2 Fermi level and redox potential of electrolyte. Thus, V_{oc} could be increased when $LiNO_3$(10)-TiO_2/FTO was used as the photoelectrode.

Y. Diamant *et al*. reported that the improvement in V_{oc} by incorporation of $SrTiO_3$ [10] and ZnO [11] onto the TiO_2 layer was attributed to a surface dipole. The formation of a surface dipole was verified by comparing the normalized IPCE curves of the DSSCs with bare and modified TiO_2. They stated that the shapes of two normalized IPCE curves were similar, such that an energy barrier was not formed in the modified TiO_2 layer. The recombination process is wavelength dependent because the penetration length of the light increases with decreasing the absorption coefficient of the dye [10,11]. N719 dye shows lower absorption coefficient at around 700 nm than its main absorption wavelength (400∼650 nm). Therefore, the photoinjected electrons originated by around 700 nm light should travel a long way to be collected by the FTO electrode. If such a distance is longer than the electron diffusion length, these electrons would be lost by recombination processes. On the other hand, if an energy barrier is formed on TiO_2 surface, the electrons photo-injected by around 700 nm light could be collected by FTO electrode because of prolonged electron lifetime induced by energy barrier, leading to a higher IPCE value. Thus, if the energy barrier is formed by surface modification, the ratio between IPCE values of DSSCs with bare TiO_2/FTO and surface-modified TiO_2/FTO, should be different at longer wavelength (around 700 nm in our case). Figure 6(a) presents the normalized IPCE curves of the DSSCs with bare TiO_2/FTO and $LiNO_3$(10)-TiO_2/FTO as the photoelectrodes. This similarity in curve shape suggests that an enhancement in V_{oc} is due to the formation of a surface dipole in the TiO_2 layer not an energy barrier.

Thus, we could conclude that the improvement in V_{oc} was caused by a conduction band edge shift of TiO_2 to the negative direction, arising from the formation of the surface dipole.

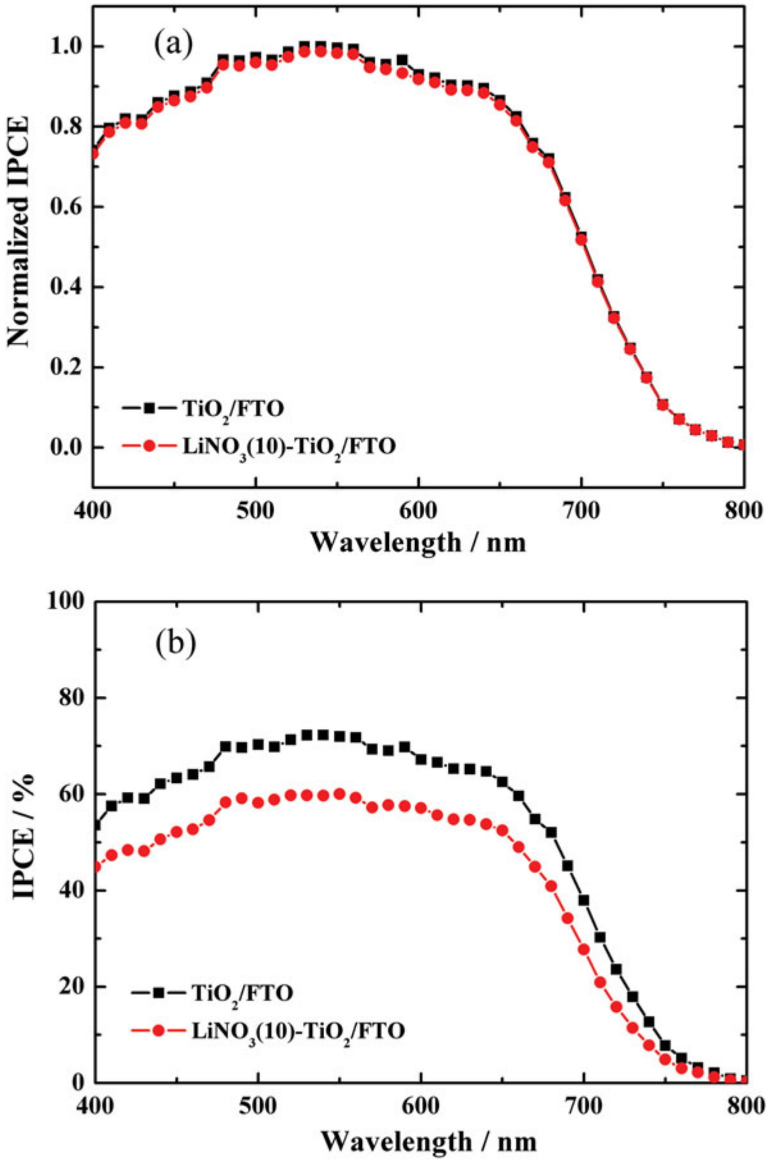

Figure 6. Normalized (a) and pristine (b) IPCE spectra for DSSCs with bare and $LiNO_3$-modified TiO_2 electrodes.

The decrease in J_{sc} was observed in the device with $LiNO_3$(10)-TiO_2/FTO electrode (Table 2). The J_{sc} value is generally influenced by four efficiency factors: light harvesting efficiency, electron injection efficiency, dye-regeneration efficiency and the electron collection efficiency of the injected electrons to the transparent electrode. In this study, the adsorbed amount of dye molecules on the $LiNO_3$-TiO_2/FTO electrode was almost similar to that of bare TiO_2/FTO electrode, as presented in Table 2, indicating that light harvesting efficiency was not changed by surface modification of TiO_2 layer. The electron collection efficiency is related to the lifetime of the electrons injected from dyes. The longer electron lifetime can increase the electron collection efficiency, owing to the reduced recombination of injected electrons to the electrolyte. It is believed that electron collection efficiency is not influenced by the surface modification, because electron lifetime was almost same in DSSCs with $LiNO_3$-TiO_2/FTO (12.148 ms) and bare TiO_2/FTO (12.147 ms). These results suggest the decrease in J_{sc} for the $LiNO_3$-modified film could be primarily related to the poorer electron injection from the excited dye to the TiO_2 film. As mentioned previously, several groups have also shown decreases in J_{sc} by employing coating materials, such as $BaCO_3$, ZnO and $SrTiO_3$, on the TiO_2 surface; the decrease is likely due to the negative shift of the conduction band edge, and this shift could cause less efficient electron injection from the excited sensitizer into the conduction band of the modified TiO_2 [10–12]. It is because the negative shift leads to a drop in potential difference between LUMO (lowest unoccupied molecular orbital) level of dye and conduction band edge of TiO_2. We therefore attribute the decreased J_{sc} to less efficient electron injection from the excited dye to the modified TiO_2 film, causing from the negative (or upward) shift of the conduction band edge by $LiNO_3$ modification. Figure 6(b) compares the IPCE spectrum of DSSC with $LiNO_3$-TiO_2/FTO to that of reference cell, which is well consistent with the decrease in J_{sc}.

Conclusions

In summary, we have modified the surface of TiO_2 photoelectrodes using aqueous $LiNO_3$ solution, and the resulting electrodes ($LiNO_3$-TiO_2/FTO) were used as the photoanode of DSSCs. The DSSC with $LiNO_3$(10)-TiO_2/FTO exhibited an increase in open circuit voltage, resulting in a PCE of 7.40%, enhanced from that (6.88%) of a reference device with bare TiO_2. By monitoring the changes in electrochemical impedance spectra, dark current-voltage characteristics and incident photon-to-current conversion efficiency, it was revealed that the modification induced the formation of the surface dipole on the TiO_2 electrode, leading to the conduction band edge shift of TiO_2 to the negative direction, and thus a larger V_{oc}.

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