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Enhancing DSSC conversion efficiency by ozone-treated TiO₂ photoanode and optimum CNT/PDDA counter electrode

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Article Info	ABSTRACT
	The conversion efficiency of dye-sensitized solar cells (DSSCs) depends on the performance of the photoanode and the counter electrode. In this paper, UV-ozone treatment has been applied to the photoanode to clean and increase the hydrophilicity of the photoanode. As a result, the dye adsorption capacity was improved. Also, low-cost multiwalled carbon nanotube (CNT) combined with poly (diallyl dimethylammonium chloride) (PDDA) was used to fabricate the counter electrode. The CNT/PDDA counter electrode was optimized to maximize its performance. By using the ozone-treated photoanode and optimum CNT/PDDA counter electrode, the conversion efficiency has increased by about 64%.

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1. INTRODUCTION

Nowadays, environmental problems caused by fossil fuels have been emerging around the world. Renewable energies replacing fossil fuel-based energies are expected to be a vital solution [1–3]. For example, wind power, hydraulic power, solar power, geothermal power. Among them, solar power is received more and more attention. Solar cells are devices that convert solar energy into electric power. The primary commercial solar cell is silicon type, which produces high conversion efficiency but high production cost. DSSC was invented in 1991 [4], which generally consists of a dye-sensitized TiO₂ thin film photoanode, an electrolyte, and a counter electrode. The photoanode is usually made by coating TiO₂ on a transparent fluorine-doped SnO₂ coated glass (FTO). DSSC has some advantages such as low production cost, less toxic manufacturing, and lightweight [5–7].

The conversion efficiency of DSSCs depends on the quality of the dyed TiO₂ photoanode and the catalytic activity of the counter electrode. The photoanode thin-film needs to be in good quality such as uniform thickness, smooth surface without cracks, and high hydrophilicity. UV-ozone treatment (ozone treatment) is known as a method for removing organic matters and improving the hydrophilicity of substrates [8, 9]. Organic matters remaining in the TiO₂ photoanode impede the dye adsorption. With the ozone treatment, the hydrophilicity of the TiO₂ photoanode is improved by forming functional groups (-COOH,-COO,-CO,-OH).

Moreover, platinum (Pt) is usually utilized to fabricate the counter electrode of DSSCs due to its high activity in redox-reaction and superior electrocatalytic activity [10–12]. Nevertheless, Pt is a rare metal and high cost [13]. Therefore, low-cost substitute materials with relatively comparable performance are needed. One promising alternative is multiwalled carbon nanotube (CNT), which has advantages of large surface area, high electrical conductivity, low cost, and chemical stability [14–16]. In addition, PDDA

(diallyl dimethylammonium chloride) has a property of electron-withdrawal from CNT. Therefore, the composite of CNT/PDDA could be used to improve the performance of the counter electrode in DSSCs [16]. The present study is focused on enhancing the conversion efficiency of the DSSC by conducting ozone treatment of the TiO_2 photoanode to improve its dye adsorption capacity, and optimizing the CNT/PDDA counter electrode. In the previous study, we have confirmed the positive effects of CNT/PDDA combination on the performance of the counter electrode of the DSSC [16]. In this paper, novel results archived by optimizing the CNT/PDDA counter electrode are presented.

2. RESEARCH METHOD
2.1. Materials

P25 TiO2 powder (particle size ~20 nm) was purchased from Aerosil Co., Ltd. Multiwalled CNT coating liquid N7006L was purchased from KJ Specialty Paper Co., Ltd., which contains 6.1 wt% multiwalled CNT with an average diameter of 9.5 nm and length of 1.5 µm [17]. 96 wt% ethanol, polyethylene glycol (PEG, average molecular 1500), 1.2-dimethyl-3-propylimidazolium iodide, toluene, and acetonitrile were purchased from Wako Pure Chemical Industries Ltd. 20 wt% PDDA solution, LiI, I_2, 4-tert-butylpyridine, and MK2 dye (2-Cyano-3-[5'''-(9-ethyl-9H-carbazol-3-yl)-3',3'',3''', 4-tetra-n-hexyl-[2,2',5',2'',5'',2''']-quarterthiophen-5-yl] acrylic acid) were purchased from Sigma Aldrich Co. LLC. Deionized water was purchased from MonotaRO Co., Ltd. All the chemicals were used as received.

2.2. Fabricating the photoanode

Compared to other thin-film coating methods such as spin coating [18], sputtering [13], doctor-blade [19], electrophoresis deposition method (EPD) has the advantages of low-cost equipment, short deposition time, high reproducibility, and controllable thickness [20]. Therefore, in this research, EPD was utilized to fabricate both the photoanodes and counter electrodes. To prepare TiO_2 colloid solution for EPD, 0.2 g P25 powder and 0.2 g PEG were mixed in 40 ml ethanol at the speed of 700 rpm for 24 h by a magnetic stirrer. An aluminum plate (20 × 20 × 1 mm) and an FTO glass (20 × 20 × 1.8 mm) were used as the EPD cathode and EPD anode, respectively. They were placed in parallel into the prepared TiO_2 solution with a 10 mm distance between the two electrodes as shown in Figure 1. A constant current of 0.12 mA from a current source (R6144, Advantest) was applied to the two electrodes. To make four-layer thin film, EPD was conducted four times with 25 s each time followed by 60ºC drying for 60 s. After that, the as-fabricated TiO_2 thin films were annealed by an electric furnace (SMF-1, Asone) at 400 ºC (5 ºC/minute heating rate) for 60 minutes. After annealing, the photoanodes were treated by a UV-ozone cleaner (UV253E, Filgen) with treating wavelength at 185 nm and 254 nm. The ozone treatment time was 24 h. The thickness of the photoanodes was about 15 µm.
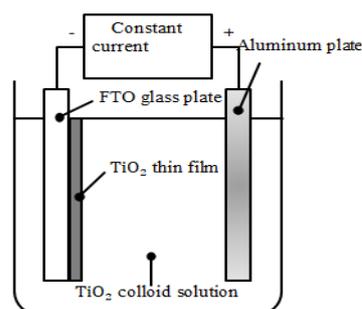
Figure 1. EPD setup for fabricating the photoanode

In DSSC research, as sensitizer dyes, ruthenium-based dyes are usually used because ruthenium dyes can absorb broad light band; as a result, they realize high conversion efficiency. However, high-cost material and environmental hazards are some problems of ruthenium-based dyes. Therefore, researchers have been trying to find low-cost and environmentally-friendly dyes for replacing ruthenium-based dyes [21–23]. In this research, organic MK2 dye was used, which is an environmentally-friendly dye. The sensitizer dye solution consisted of 10 mg MK2 dye dissolved in 108 ml toluene. The prepared TiO_2 photoanodes were soaked in the dye solution at 25 ºC for 3 h. After that, their surfaces were rinsed by toluene and deionized water continuously. Then, the dyed photoanodes were dried at 80 ºC in a drying oven for 10 minutes.

2.3. Fabricating the counter electrode

To fabricate the counter electrode, an aluminum plate (40×20×1 mm) and an FTO glass (40×20×1.8 mm) were used as the EPD anode and EPD cathode, respectively. The two electrodes were set at 9 mm distance as shown in Figure 2. Constant 3 V from a power source (AD-8723D) was applied on the two electrodes and EPD time was 90 s. First, the counter electrode using only CNT (CNT coating liquid was used directly for EPD) was fabricated. After EPD the as-fabricated counter electrode was dried at room temperature for 24 h. This counter electrode is called the CNT counter electrode. Second, the CNT/PDDA counter electrode was fabricated. The preparation flow of the CNT/PDDA solution used for EPD is shown in Figure 3. 20 wt% PDDA solution was diluted in deionized water to obtain 1 wt% PDDA solution. After that, CNT coating liquid and 1 wt% PDDA solution were mixed with five different ratios of CNT/PDDA=5~15/1 to obtain five CNT/PDDA solutions for conducting EPD. The EPD process was the same as above. The as-fabricated CNT/PDDA counter electrodes were dried at room temperature for 24 h. This counter electrode is called CNT/PDDA counter electrodes. The thickness of these counter electrodes was in the range of 10~13 µm.
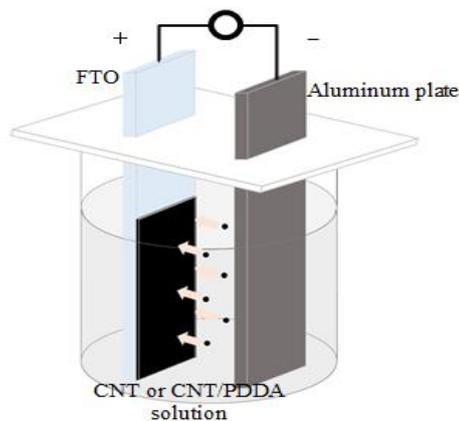
Figure 2. EPD setup for fabricating the counter electrode
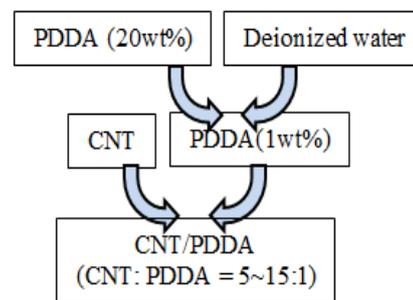
Figure 3. The preparation flow of CNT/PDDA solutions used for fabricating CNT/PDDA counter electrodes

2.4. DSS construction

In this research, we used the acetonitrile solvent-based electrolyte, which consisted of 0.6 M 1.2-dimethyl-3-propylimidazolium iodide, 0.1 M LiI, 0.2 M I_2, and 0.5 M 4-tert-butylpyridine. The DSSC was assembled using the two fabricated electrodes and the electrolyte. As shown in Figure 4, masking tape made of 3M tape (~ 45 µm thickness) was cut a 25 mm^2 square hole, then adhered to the photoanode. A suitable amount of the electrolyte was dropped on the active area, and the counter electrode was put on top of this area. Two binder clips were used to clamp the cell for measurement. In this work, we focused on finding the optimum conditions of the photoanode and counter electrode for improving the conversion efficiency of the DSSC. To save time and lower the cost of this research, the DSSC was made for short-time measurement only.
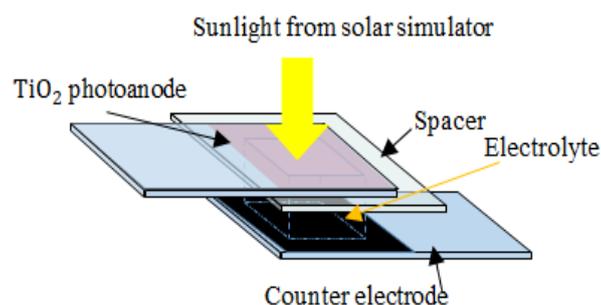
Figure 4. DSSC construction

2.5. Characterization method

The thickness of the photoanode and counter electrode were measured by a step gauge (Bruker DektakXT). The photoanode surface was observed by a scanning electron microscope (SEM, Hitachi S4300). The absorbance spectrum of the photoanode was measured by a spectrophotometer (Shimadzu UV-3600). The open-circuit voltage (V_{oc}), short-circuit current density (J_{sc}), fill factor (FF), and conversion efficiency (η) of the fabricated DSSC were measured with a solar simulator (OAI TriSOL) simulating the sunlight (AM1.5, 100 mW/cm^2). For each experiment, four samples were measured, and the average result was presented in this paper. Error bars in the presented figures were based on the results of the measured samples.

3.   RESULTS AND ANALYSIS

3.1. Photoanode characterization

The surface of the annealed TiO$_2$ thin film was observed by SEM. Figure 5 shows the surface of the four-layer TiO$_2$ thin film. Almost no crack was observed on the surface. It has been proved that multilayer EPD method could significantly improve the quality of thin films [24-26]. Figure 6 shows the photo images of the dyed photoanodes with and without ozone treatment. The photoanode with the ozone treatment had a darker color than the photoanode without the ozone treatment. In other words, the photoanode with the ozone treatment had adsorbed more dye than the photoanode without the ozone treatment. The ozone treatment removed remaining organic matters and improves the hydrophilicity of the photoanode surface and therefore facilitated the adsorption of MK2 dye.
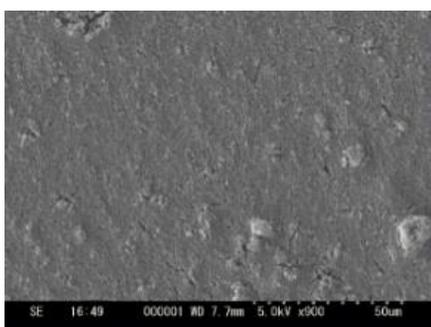
Figure 5. An SEM image of the surface of the photoanode after annealing

Figure 6. The photo images of the dyed photoanodes with the ozone treatment (left) and without the ozone treatment (right)

UV-vis absorbance spectra of the dyed photoanodes with and without the ozone treatment were also measured. As shown in Figure 7, MK2 dye enabled visible light absorbance for the photoanodes with the maximum absorbance obtained around 480 nm. The photoanode with ozone treatment had a better UV-vis absorbance spectrum in the range of 400-800 nm with significantly higher absorbance and a larger spectrum. From this result, it can be confirmed that the ozone treatment can improve the absorbance characteristic of the photoanode.
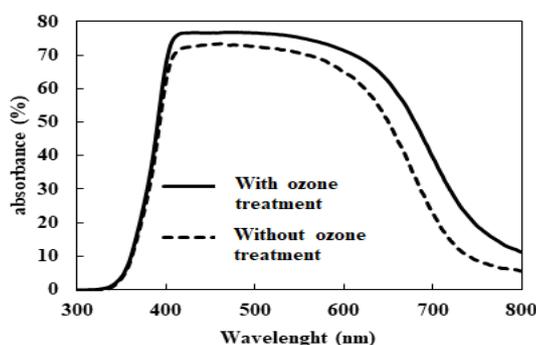
Figure 7. UV-vis absorbance spectra of the dyed photoanodes with and without the ozone treatment

3.2. Counter electrode optimization

In this subsection, the performance of various counter electrodes in the DSSC was investigated. All experiments in this section used the photoanodes without ozone treatment. First, the CNT and CNT/PDDA counter electrodes were compared. The measured photovoltaic parameters of the DSSCs using the counter electrodes made of CNT and CNT/PDDA (CNT/PDDA=10:1) are listed in Table 1. The DSSC using the CNT counter electrode generated 2.78% conversion efficiency while the DSSC using the CNT/PDDA counter electrode generated 4.12% conversion efficiency. This was about 48% improvement. This improvement was attributed to the presence of PDDA in the counter electrode [16].

Table 1. Photovoltaic parameters of the DSSCs made with two different types of counter electrodes: CNT and CNT/PDDA (CNT/PDDA=10:1)

Counter electrode	Jsc (mA/cm^2)	Voc (V)	FF (%)	η (%)
CNT	10.31	0.67	40.62	2.78
CNT/PDDA	12.82	0.70	45.64	4.12

Second, the optimization of the CNT/PDDA counter electrode was investigated. Figure 8 shows the conversion efficiency of the DSSC as a function of the CNT/PDDA ratio. As the ratio was varied from 5:1 to 15:1, the conversion efficiencies were 3.58, 3.75, 4.12, 3.48, and 3.17%, respectively, as listed in Table 2. The maximum conversion efficiency was achieved at the ratio of CNT/PDDA=10:1. Therefore, we used this ratio as the optimum ratio of the CNT/PDDA counter electrode.
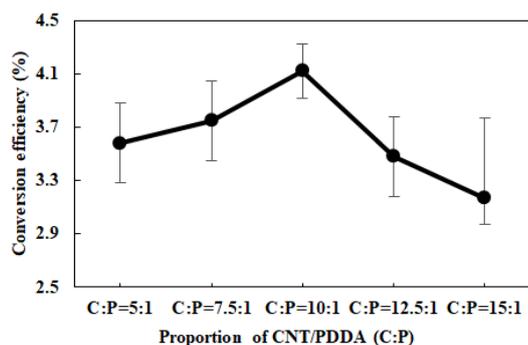
Figure 8. The conversion efficiencies of the DSSCs using CNT/PDDA counter electrodes with different CNT/PDDA ratios. Error bars represent standard deviation

Table 2. Photovoltaic parameters of the DSSCs using CNT/PDDA counter electrodes with different CNT/PDDA ratios

CNT:PDDA	Jsc (mA/cm^2)	Voc (V)	FF (%)	η (%)
5:1	11.81	0.68	44.41	3.58
7.5:1	11,80	0.70	45.26	3.75
10:1	12.82	0.70	45.64	4.12
12.5:1	11.09	0.69	45.42	3.48
15:1	10.45	0.66	45.83	3.17

3.3. Effectiveness of the ozone-treated photoanode on the DSSC performance

The DSSCs using the optimum CNT/PDDA counter electrodes (CNT/PDDA=10:1) and the photoanodes with and without the ozone treatment were measured. Figure 9 displays the J-V curves of these two DSSCs, and Table 3 lists their measured photovoltaic parameters. Compared with the DSSC using the photoanode without the ozone treatment, the DSSC using the photoanode with the ozone treatment showed the increase in Jsc (from 12.82 to 14.12 mA/cm^2) and FF (from 45.64 to 49.39%). As a result, about 11% enhancement in the conversion efficiency was obtained (from 4.12 to 4.57%). This result confirmed the positive effect of the ozone treatment on the performance of the photoanode. If compared with the DSSC using the CNT counter electrode and the photoanode without the ozone treatment, the DSSC using the optimum CNT/PDDA counter electrode and the photoanode with the ozone treatment showed 64% improvement in the conversion efficiency (4.57% compared with 2.78%).
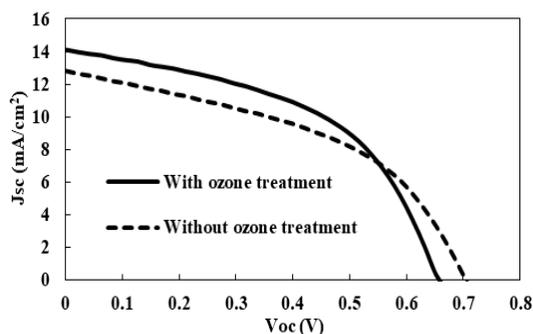
Figure 9. The J-V curves of the DSSCs using the photoanodes with and without the ozone treatment and the optimum CNT/PDDA counter electrodes (CNT/PDDA=10:1)

Table 3. Photovoltaic parameters of the DSSCs using the photoanodes with and without the ozone treatment and the optimum CNT/PDDA counter electrodes (CNT/PDDA = 10:1)

Photoanode	Jsc (mA/cm^2)	Voc (V)	FF (%)	η (%)
Without ozone treatment	12.82	0.70	45.64	4.12
With ozone treatment	14.12	0.67	49.39	4.57

4. CONCLUSION

In this study, the DSSC conversion efficiency was improved by optimizing the counter electrode and using the photoanode with ozone treatment. The CNT/PDDA counter electrode has been optimized to maximize the DSSC conversion efficiency. In addition, the ozone treatment improved the dye adsorption of TiO_2 thin film photoanode and increased the conversion efficiency of the DSSC by about 11%. From the experimental results, the conversion efficiency of the DSSC was successfully improved by about 64% by using the ozone-treated photoanode and the optimum CNT/PDDA counter electrode.

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