

Research Article

Evaluation and Optimization to Recycle Used TiO₂ Photoelectrode for Dye-Sensitized Solar Cells

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This study proposes a method for recycling and activating the titanium oxide (TiO₂)/fluorine-doped tin oxide (FTO) photoanode in dye-sensitized solar cells (DSCs) by repeated dye adsorption and desorption processes using various desorption agents. This simple and convenient method could be utilized to activate TiO₂ photoelectrodes for DSCs after the long-term operation. The devices are immersed in acidic, alkaline, and neutral media of various concentrations for desorption and then are soaked in the N719 solution again. The optimal device had an overall power conversion efficiency (AM 1.5 G, 100 mW/cm²) with 5 × 10⁻³ M NaOH solution as a desorption agent being 6.44% better than that of devices that had not undergone recycling and activation.

1. Introduction

Since O'Regan and Grätzel successfully proposed dye-sensitized solar cells (DSCs) using nanocrystalline structures in 1991 [1], research on such cells has intensified. Most of the extensive literature on DSCs has focused on improving the light to electricity conversion [2, 3]. Nevertheless, the ultimate goal is to develop commercial applications. For practical purposes, DSC modules must have long-term stability and function under long-term irradiation because up to millions of electrons are generated from sensitizer molecule [4]. However, some modules may age [4–6] by breaking or desorbing dye molecules from the titanium oxide (TiO₂) surface arising from the H₂O in the atmosphere after long-term exposure to full sunlight [7]. Furthermore, the transparent conductive electrode, which is an important component of a DSC, is also expensive [8–10]. Accordingly, DSC modules should have the repairable or recyclable functionalities. Relatively little effort has so far been made to investigate these ageing modules following long-term operation. Therefore, this work proposes a new method for recycling photoanodes with the purpose of recycling and activating commercially available DSC modules that have decayed through use by rinsing off the dye molecules from the TiO₂ and reusing the photoanode. The photoanode can be reused for the uptake of dye in a new

module, reducing manufacturing costs by eliminating the need to fabricate new substrates. After recycling, the original dye molecules in the used DSC modules can be replaced by the newly synthesized Ru complex photosensitizer or organic dyes with a higher conversion efficiency.

We previously demonstrated a novel concept of extending the lifetime of dye-sensitized solar cells (DSCs) and reducing the costs of reconditioning them by recycling FTO/TiO₂ substrates [11]. The results revealed that dye adsorption and V_{oc} under multiple dye adsorption/desorption processes were significantly increased, improving the power conversion efficiency. However, we did not elucidate the influence of using various concentrations of desorption agents on the DSCs. Hence, this work develops a unique, simple, and convenient method of activating the photoelectrode using alkaline, acidic, and neutral solutions as desorption agents. After the concentrations of desorption solution are optimized for desorption in the recycling and activating processes, the DSC is fabricated with the TiO₂ substrate using 5 × 10⁻³ M NaOH as a desorption agent, yielding a V_{oc} of 0.78 V, a J_{sc} of 10.79 mA/cm², and a fill factor (FF) of 0.76, corresponding to a power conversion efficiency (PCE, η) of 6.44% (AM 1.5 G, 100 mW/cm²). The results verify the possibility of recycling and reactivating DSC modules.

2. Experimental Details

2.1. Materials. The Ru dye, *cis*-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II), bis-tetrabutylammonium ($\text{RuL}_2(\text{NCS})_2\text{-2TBA}$, also known as N719), and fluorine-doped SnO_2 conductive glass (FTO, $8 \Omega/\text{square}$) were purchased from Solaronix. The TiO_2 paste (ETERDSC Ti-2105) was provided from ETERNAL CHEMICAL CO., LTD. Sodium hydroxide (NaOH) and hydrochloric acid (HCl) were purchased from Acros. Acetic acid (CH_3COOH) and sodium acetate (CH_3COONa) were purchased from J. T. Baker.

2.2. Fabrication of DSCs. The dye-sensitized solar cells were fabricated as follows. The TiO_2 paste of 25 nm particle size was cast on the FTO by the doctor-blading method using an adhesive tape as the spacer, followed by sintering at 495°C for 30 minutes in air to obtain a transparent TiO_2 photoelectrode of an effective area of 1 cm^2 with the thickness of $11 \mu\text{m}$. The mesoporous TiO_2 photoelectrode was immersed in the N719 solution of $5 \times 10^{-4} \text{ M}$ in acetonitrile and tert-butanol (volume ratio 1:1) for 18 hrs to complete the sensitizer loading. The cells were assembled by dropping an electrolyte containing 0.1 M LiI, 0.05 M I_2 , 0.6 M 1-propyl-2,3-dimethylimidazolium iodide (DMPII), 0.5 M tert-butylpyridine in 3-methoxypropionitrile, and a platinum sheet as a counter electrode. For the desorption and activation of N719 dye molecules from TiO_2/FTO substrate, the dyed TiO_2 films were immersed in 10^{-2} M , $5 \times 10^{-3} \text{ M}$, 10^{-3} M , and 10^{-5} M of different solutions as desorption agents (NaOH [12–14], CH_3COONa , HCl [15], and CH_3COOH) in a mixture of ethanol and deionized water (DI water) with 1:1 volume ratio separately. The TiO_2/FTO substrates after immersing in desorption agents were immersed in the N719 solution for 18 hrs and assembled again. The following desorption/adsorption processes were repeated with the same procedure.

2.3. Characterization. The devices were characterized by a Keithley 2400 sourcemeter using a metal mask with an aperture area of 0.25 cm^2 under standard AM 1.5 G filtered irradiation ($100 \text{ mW}/\text{cm}^2$) from a Newport M-91190A Solar Simulator. UV-Vis spectra were measured by a spectrometer (U-3310, Hitachi). The images of the TiO_2 film were characterized by Field-Emission Scanning Electron Microscopy (FESEM, JSM-6701F). The crystallographic properties of TiO_2 were characterized by X-ray diffraction microscopy (Rigaku D/MAX2500) at scan rate of $4^\circ/\text{min}$.

3. Results and Discussion

3.1. Variation of Desorption Process Using Alkaline, Acidic and Neutral Solutions. Figure 1 presents the UV-Vis absorption spectra of dyed TiO_2 films following the various desorption processes. Alkaline, acidic, and neutral solutions were compared for effectiveness as agents for desorbing dye molecules from surface of the TiO_2 . Figure 1(a) displays the UV-Vis absorption spectra of the dyed TiO_2 films, measured after they were immersed in 10^{-2} M solutions of NaOH,

TABLE 1: pH values of desorption agents.

	1 M	10^{-2} M	$5 \times 10^{-3} \text{ M}$	10^{-3} M	10^{-5} M
NaOH	13.00	12.39	12.29	11.59	7.55
CH_3COONa	8.50	7.71	7.62	7.6	6.52
HCl	0.43	2.12	2.37	2.93	4.99
CH_3COOH	2.98	4.13	4.20	4.39	5.30

CH_3COONa , HCl, and CH_3COOH in deionized water (DI water) with ethanol solutions (volume ratio 1:1) for two minutes, as well as the spectrum of bare TiO_2 . In Figure 1(a), the maximum absorption peak of the untreated dyed TiO_2/FTO substrate is around 450~600 nm, associated with metal-to-ligand charge-transfer (MLCT) transition [16, 17]. The MLCT band of the dyed TiO_2/FTO substrate completely disappeared when it was immersed in NaOH solutions, but its intensity was slightly higher than that of bare TiO_2 with CH_3COONa solution as the desorption agent. This observation suggests the total removal of dye molecules from TiO_2 by NaOH solutions, but a few dye molecules remained on TiO_2 by CH_3COONa solution. When acid agents desorb dye molecules from TiO_2 , the intensity of the MLCT band is one-third lower than that of the bare TiO_2 after immersion in HCl and CH_3COOH solutions, respectively. Accordingly, 10^{-2} M HCl and CH_3COOH solutions are not effective agents for detaching N719 dye molecules from TiO_2 but NaOH and CH_3COONa solutions are effective. In addition, the intensity of the MLCT band dropped to only half upon immersion in DI water with ethanol as compared to that of the bare TiO_2 . The above results reveal that immersion in these solutions at a given concentration changes the MLCT band in significantly different ways. These results suggest that the desorption process depended on the pH value of the solutions. Hence, the optimal concentration of these desorption agents is sought to desorb dye molecules effectively from TiO_2 .

Figures 1(b), 1(c), 1(d), and 1(e) show the UV-Vis absorption spectra of the dyed TiO_2 films following immersion in NaOH, CH_3COONa , HCl, and CH_3COOH solutions with various concentrations. Table 1 presents the pH values of these solutions. In Figure 1(b), the adsorbed dye molecules were completely desorbed from the TiO_2 surface using NaOH solution at a concentration of 10^{-2} M or $5 \times 10^{-3} \text{ M}$ as the desorption agent. Nevertheless, most dye molecules remained on the TiO_2 when the concentration exceeded $5 \times 10^{-3} \text{ M}$. In Figure 1(c), the intensity of MLCT band gradually decreased as the concentration of the CH_3COONa solution increased. However, the degree of desorption in the 10^{-2} M CH_3COONa solution was still lower than in the $5 \times 10^{-3} \text{ M}$ NaOH solution. Figure 1(a) presents that the use of 10^{-2} M acid desorption agent for two minutes resulted in poor desorption. Thus, the immersion time was extended to 16 hours using acid solution as the desorption agent. The intensity of MLCT band decreased as the concentrations of the HCl or CH_3COOH solutions decreased, as shown in Figures 1(d) and 1(e). This finding indicates that acid inhibited desorption. As noted above, the optimal concentrations and immersion times of

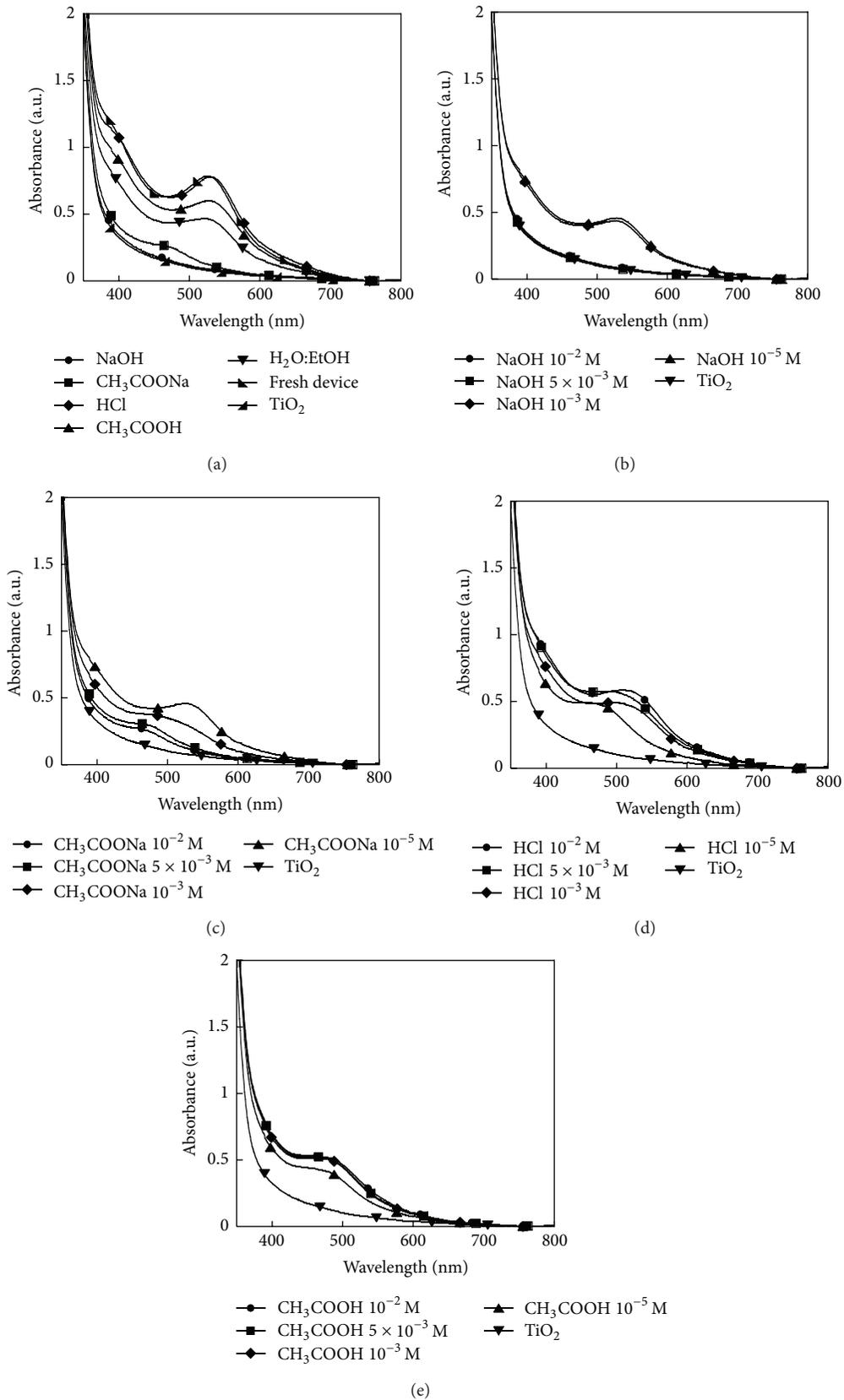
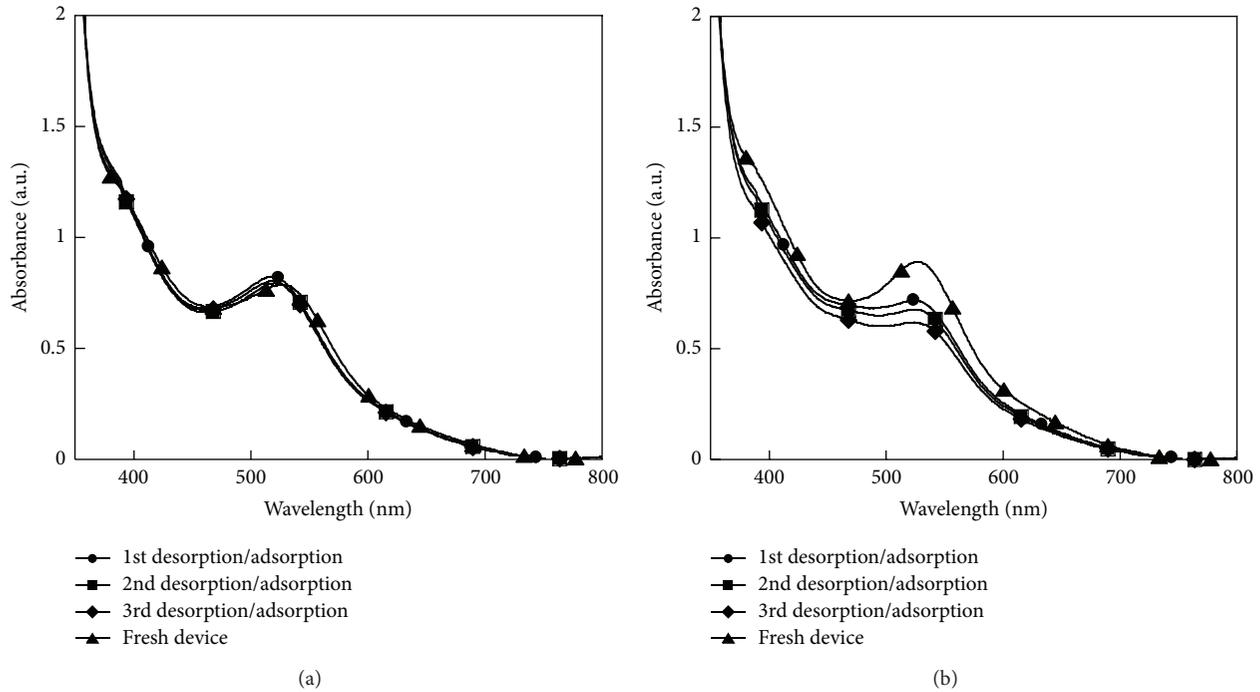


FIGURE 1: (a) UV-Vis absorption spectra of TiO₂ substrates after desorption using NaOH, CH₃COONa, HCl, CH₃COOH, and DI water with ethanol solutions. UV-Vis absorption spectra of dyed TiO₂ films after immersion in (b) NaOH and (c) CH₃COONa for two minutes and (d) HCl and (e) CH₃COOH solutions for sixteen hours at various concentrations.

TABLE 2: The photovoltaic performance of DSCs under multiple adsorption/desorption processes using NaOH solution as desorption agent.

Device	V_{oc} (V)	J_{sc} (mA/cm ²)	FF	Efficiency (%)
Fresh device	0.74	10.79	0.77	6.22
1st desorption/adsorption	0.76	10.87	0.75	6.23
2nd desorption/adsorption	0.78	10.79	0.76	6.44
3rd desorption/adsorption	0.77	10.81	0.74	6.19

FIGURE 2: UV-Vis absorption spectra of dyed TiO₂ following readsorption of N719 dye using (a) 5×10^{-3} M NaOH and (b) 10^{-5} M CH₃COOH as desorption agents under multiple adsorption/desorption processes.

the solutions for desorption were 5×10^{-3} M NaOH for two minutes and 10^{-5} M CH₃COOH for 16 hours. Thus, to verify the variation of the readsorption of the dye after immersion in desorption agents, the DSCs were measured during multiple adsorption/desorption processes using these two solutions, as described in the following section.

3.2. Variation of Multiple Adsorption/Desorption Process Using Alkaline and Acidic Solutions. Figure 2 shows the UV-Vis absorption spectra of the dyed TiO₂ film following readsorption of N719 dye using NaOH and CH₃COOH as desorption agents. Notably, after readsorption using NaOH as a desorption agent, the intensity of the MLCT band of the TiO₂ substrate was slightly increased, as can be observed in Figure 2(a), indicating an increase in the adsorption of dye on the TiO₂ film. However, the readsorption by the TiO₂ substrate after being desorbed by CH₃COOH is significantly less than adsorption by a fresh device, because of the strong anchoring capabilities of the carboxylate groups in CH₃COOH, which occupy some of the active sites on the TiO₂ surface, and the subsequent decrease in the adsorption of dye molecules [18].

3.3. The Photovoltaic Performance of DSCs for Multiple Adsorption/Desorption Using Alkaline and Acidic Solutions. Figure 3 plots the current-voltage (J - V) characteristic curves of the fresh device and those that have undergone multiple adsorption/desorption processes with NaOH and CH₃COOH solutions as desorption agents. Tables 2 and 3 present their detailed photovoltaic parameters. Figures 3(a) and 3(b) present the photovoltaic performance of DSCs under multiple adsorption/desorption processes using NaOH and CH₃COOH solutions as desorption agents, respectively. In Figure 3(a), for the fresh device, V_{oc} was 0.74 V, J_{sc} was 10.79 mA/cm², FF was 0.77, and η was 6.22%. In the 1st desorption/adsorption device with NaOH solution, the J_{sc} , V_{oc} , FF and η were 0.76 V, 10.87 mA/cm², 0.75, and 6.23%, respectively, which are comparable to those of a fresh device. However, the 2nd desorption/adsorption device had a higher power conversion efficiency of 6.44%, which is higher than that of the 1st desorption/adsorption device because of its higher V_{oc} of 0.78 V. Although the FF, and η of the 3rd desorption/adsorption device are a little lower than those of the fresh device, its V_{oc} is markedly increased from 0.74 to 0.77 V. The increase in V_{oc} may result from the suppression of electron back-transfer [19–21] or upward shift of the TiO₂

TABLE 3: The photovoltaic performance of DSCs under multiple adsorption/desorption processes using CH_3COOH solution as desorption agent.

Device	V_{oc} (V)	J_{sc} (mA/cm^2)	FF	Efficiency (%)
Fresh device	0.74	10.83	0.78	6.24
1st desorption/adsorption	0.62	5.89	0.74	2.71
2nd desorption/adsorption	0.56	2.90	0.71	1.16
3rd desorption/adsorption	0.54	1.67	0.66	0.60

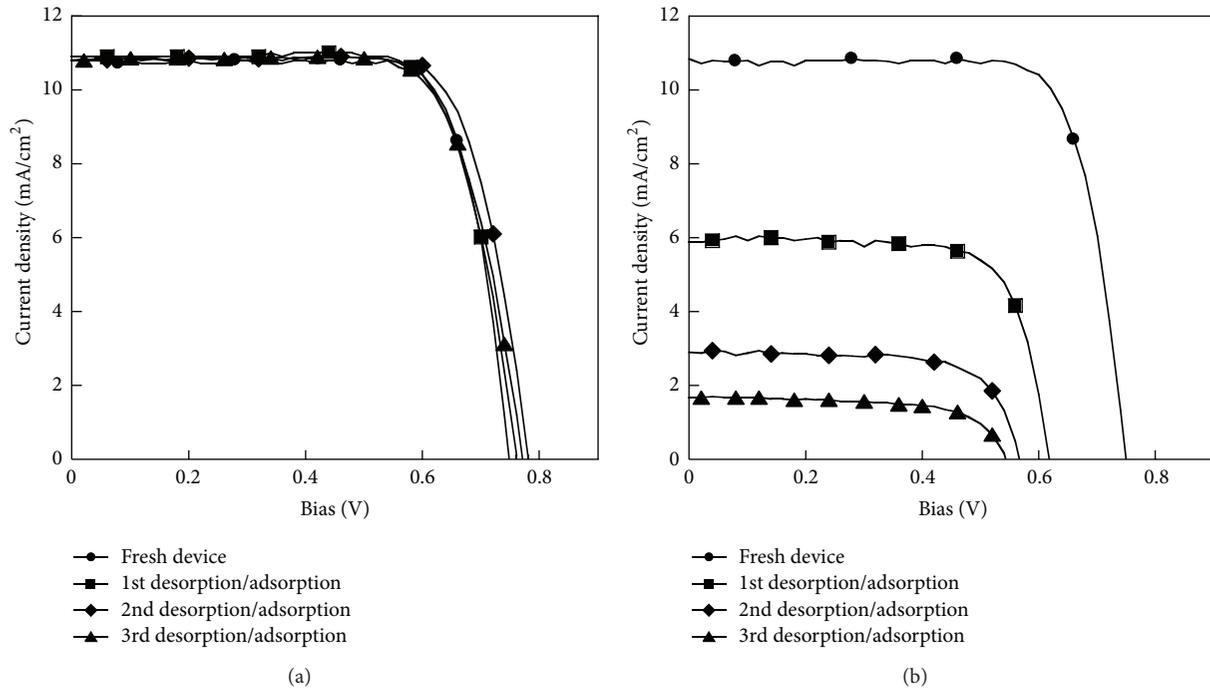


FIGURE 3: J - V characteristic curves of fresh DSCs and devices that had undergone multiple adsorption/desorption processes using (a) NaOH and (b) CH_3COOH solutions as desorption agents under AM 1.5 G, $100 \text{ mW}/\text{cm}^2$ illumination.

conduction band [22, 23] relative to the electrolyte potential. In Figure 3(b), the desorption/adsorption devices were all worse than the fresh device. The poor performance may be ascribed to the decrease of light harvesting that resulted from a decreased amount of dye corresponding to Figure 2(b). Another reason may result from the binding mode between dye and TiO_2 that favors a “carboxylic acid” type linkage while it favors photoelectrode in high proton concentrations. The “carboxylic acid” linkage is easier to desorption than the “carboxylate” type linkage when Li^+ is present [24]. Thus, CH_3COOH solution is not an effective desorption agent.

3.4. The Surface Properties of DSCs for Adsorption/Desorption Using Alkaline Solutions. To confirm the surface properties of TiO_2 under multiple adsorption/desorption processes, FESEM was used to determine their morphologies. Figure 4 presents the FESEM images of the bare TiO_2 film (Figure 4(a)) and the TiO_2 film that had undergone desorption using $5 \times 10^{-3} \text{ M}$ NaOH (Figure 4(b)) as the desorption agent. The bare TiO_2 films and the films after immersion in NaOH solution differed only slightly in surface topography, indicating that the morphology of TiO_2 films did

not significantly change upon desorption by NaOH solution. However, if the concentration of NaOH solution becomes excessive, then the surface of TiO_2 films may be damaged. Figures 4(c) and 4(d) display the bare TiO_2 film and the film after the use of 5 M NaOH solution as a desorption agent, respectively. At higher concentrations, some cracks appear on the surface. The results prove that NaOH solution at a low concentration is an effective agent for desorption.

Furthermore, the XRD measurements were made to determine whether immersion in various desorption agents changed the crystallinity of TiO_2 . Figure 5 presents the XRD patterns of the TiO_2 film desorbed by various solutions. In Figure 5, the structure of the bare TiO_2 film is anatase, which is the same as that of the other TiO_2 films that have undergone desorption using various solutions as desorption agents. The figure reveals that desorption processes did not change the crystallinity. This proposed concept verifies the influence on DSCs using various desorption agents and enhances its performance. This method may be able to apply in the prototype DSC modules. Figure 6 shows the photo of the prototype reusable DSC modules. The detailed results about this experiment will be discussed in the following paper.

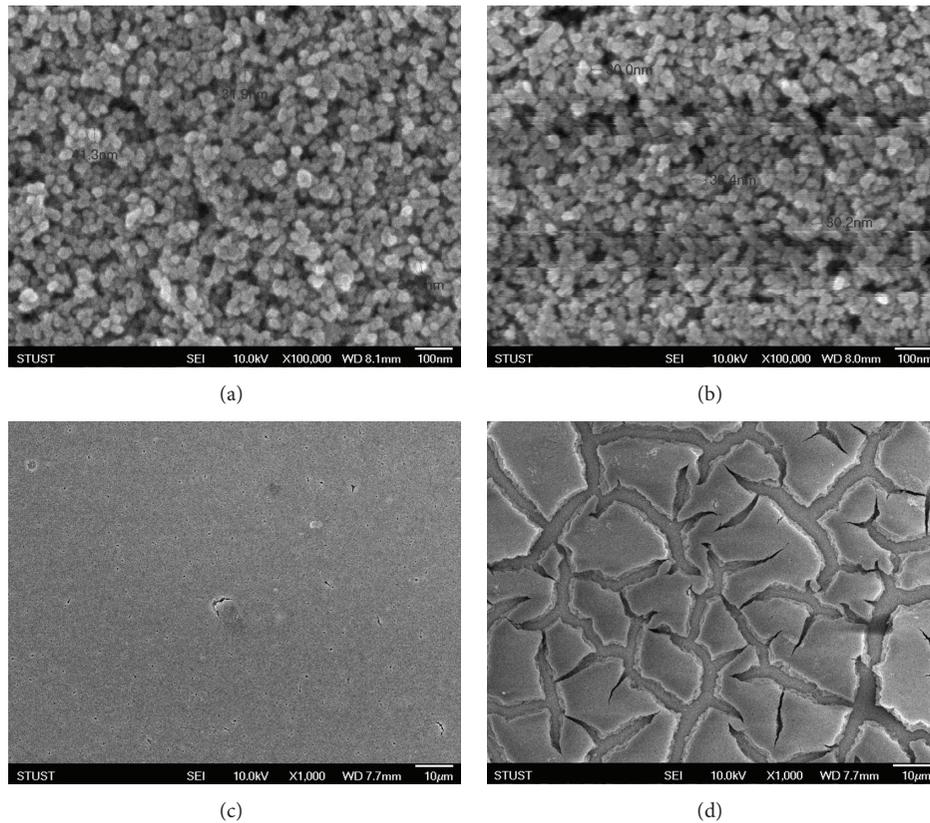


FIGURE 4: FESEM images of ((a), (c)) bare TiO_2 film and TiO_2 film after use of (b) 5×10^{-3} M and (d) 5 M NaOH solutions as desorption agents.

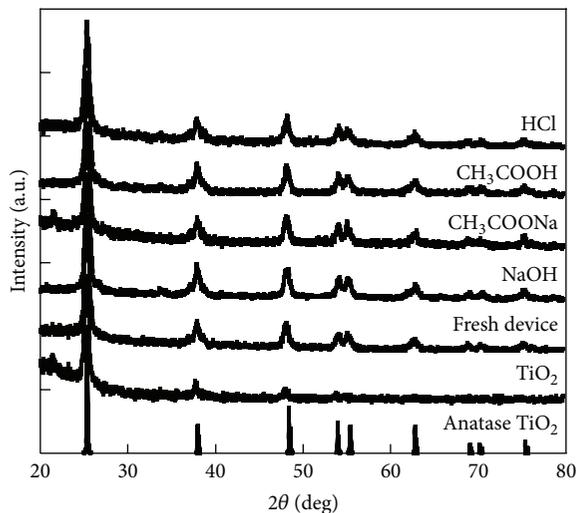


FIGURE 5: X-ray diffraction patterns of TiO_2 film that has undergone desorption by NaOH, CH_3COONa , HCl, and CH_3COOH solutions.

4. Conclusion

In summary, a TiO_2 surface was successfully recycled and activated using alkaline, acidic, and neutral solutions as desorption agents. The degree of desorption using NaOH

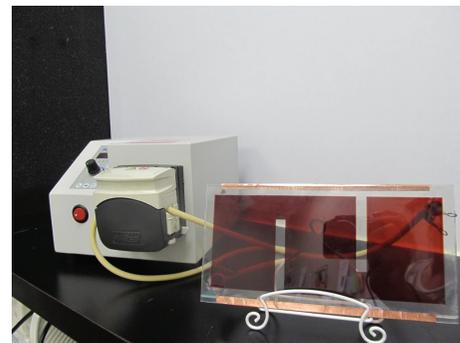


FIGURE 6: The photo of the prototype reusable DSC modules.

exceeded that using acid and neutral solutions. The enhancement in J_{sc} and V_{oc} upon desorption may have been caused by the increase in loading density of dye molecules on the TiO_2 films and the suppression of the back electron transfer, respectively. The highest obtained η was 6.44%, which was obtained for the recycled DSCs following two minutes of desorption using 5×10^{-3} M NaOH. This work demonstrates the feasibility of reusing a photoelectrode of DSC modules after long-term operation. The advantages of this method are that it reduces the cost of reconditioning DSC modules and improves their performance. This research would be of great

interest if the newly synthesized photosensitizers with the higher conversion efficiency were used in place of the original dye molecules in the used DSC modules after a long period of operation.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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