Photoelectrochemical Characteristics of N-and S-doped TiO$_2$ with Coumarin

Chiu-Hsuan Lee, Je-Lueng Shie, Ching-Yi Tsai, and Ching-Yuan Chang

Abstract—**This study investigated the photoelectronchemical characteristics of dye-sensitized N- and S-doped TiO$_2$ (TiN$_x$O$_{2-x}$S$_y$) with coumarin using visible light emitting diode (VLED).N- and S-doped TiO$_2$was prepared bytitanium sulfate (Ti(SO$_4$)$_2$) and ammonia (NH$_3$). TiO$_2$ and TiN$_x$O$_{2-x}$S$_y$ films on indium-doping tin oxide (ITO) promoted with nature organic sensitizers of coumarin (C$_9$H$_6$O$_2$) were prepared by precipitation method. The HRSEM, UV/Vis absorption spectra, Elemental Analyses andBET surface area of dye-sensitizedN- and S-doped TiO$_2$ as well as I-V curve test of TiN$_x$O$_{2-x}$S$_y$ solar cell (TNSSC)dye-sensitizedwith and without coumarin (CONSDSSC and CODSSC, respectively)under the irradiation of visible light lamp (VLL),blue light emitting light (BLED), white LED (WLED)andred LED (RLED) were measured.All the values of I-V curve ofCONSDSSC are larger than those of CODSSC. The maximum power (P$_{max}$) was 4.81 µWfor CONSDSSC under VLL and it was larger than that of CODSSC under VLL almost 17.8 times.This result shows that the TiN$_x$O$_{2-x}$ solar cell dyedwith coumarin can largely increase the photoelectronchemical characteristics and the absorption light wavelength is changedto visible light range with red shift obviously. All the results obtained from this study can apply for the design of indoor dye sensitized solar cell (DSSC) with indoor light sources.**

Index Terms—**Dye sensitized solar cell (DSSC), coumarin, light emitting diode (LED), N-doped TiO$_2$, photoelectrochemical characteristics.**

I. INTRODUCTION

Interest in dye-sensitized solar cells (DSSCs) since the first introduction of high surface area TiO$_2$ nanoparticles as a photo-anode has focused on improving the photo-electricity conversion efficiency for commercial products [1]. DSSCs have been attracting considerable attention owing to their comparatively low cost and high efficiency in recent years [2]. A DSSC is essentially aphotoelectrochemical system, in which the light harvesting is accomplished by dye molecules that are adsorbed on the surface of the oxide nanostructures that form the photoelectrode film. During the operation process, photons captured by the dye monolayer create excitations that are rapidly split at the nano crystallite surface of the oxide film. Electrons are injected into the oxide film and holes are released by the redox couples in the liquid electrolyte. Dyes are the crucial factor for the solar to energy efficiency of DSSCs. There are several types of properties related with dyes which determine the efficiency of DSSCs, such as sunlight harvesting efficiency, quantum yield of electron injection on the surface of semiconductor film, transparent conducting oxide substrate, photoanode, light absorber, redox electrolyte and counter electrode, photo-response wave length etc. [3], [4].The various approaches using the light scattering effect associated with light absorbers have been surveyed and investigated to increase the light absorption to longer wavelength [5], [6].

Improvement in the light harvesting efficiency (LHE) of the photoelectrodes of DSSCs and molecular modification of dyes are tough task for their complicated synthesis routes. Polybipyridyl ruthenium dyes are the most efficient photo sensitizer in DSSCs by now [7]. However, the cost of dye needs to be considered. One way to improve the LHE of a photoelectrode is a use of new and cheap sensitizers with the increased absorption coefficients and/or the extended long-wavelength absorption edges. Such an approach has led to significant improvements in the conversion efficiency of DSSCs. Using coumarine or polyene type sensitizers, strikingly high solarto electric power conversion efficiencies reaching up to 7.7%in full sunlight have been achieved. With the dye sensitized system, more of the incident solar radiation would be available to activate the TiO$_2$ resulting in accelerating the photo-oxidation. However, sunlight is not easy available in indoor or dark night, high reaction rates require intense light in the appropriate spectral region [8], [9]. Asahi *et al*. [10] found that nitrogen-doped TiO$_2$ possesses visible light activity. The nitrogen-doped TiO$_2$ was prepared under the atmosphere of NH$_3$ gas using commercial TiO$_2$.There have been many other reports [11]-[16] on the introduction of N atoms through substitutional doping into the TiO$_2$ lattice in place of O atoms, mainly to decrease the TiO$_2$ band gap (3.2 eV) and henceto improve the associated photocatalytic activity under visible light. Additionally, N-doped TiO$_2$also can be prepared directly in the calcinationsprocess of TiO$_2$ using the method of neutralization hydrolyzation [17]. It has been reported that nitrogen-doped TiO$_2$ is visible light active for photocatalytic hydrogen evolution [18], but its activity is not high enough. However, there had no discussions on the N-doping process combined with organic dye of coumarin for DSSCs and also no evaluation of the N-doped DSSCs with the light sources of LED for Building-integrated photovoltaic (BIPV). Dyes are best used with sources well-matched to their absorption spectra, which can be achieved with a choice of a high intensity LED [19]-[24], especially in indoor lighting circumstance. In this study, we mainly evaluated the characteristics of N-dopedTiO$_2$ films sensitized by coumarin under the irradiation of visible light sources.

II. MATERIALS AND METHODS

A. Coumarin and Dye-Sensitized Catalyst

In this work, coumarin ($C_9H_6O_2$) dye was used as a photosensitizer of the catalysts. N-and S-doped TiO_2 ($TiN_xO_{2-x}S_y$) was prepared by the incipient wet impregnation method with $Ti(SO_4)_2$ (20 wt. %) solution which was adjusted to PH = 7 and concentration of SO_4^{2-} less than 0.5 mg L^{-1} with aqueous ammonia (28 wt. %). After being filtered and washed to remove SO_4^{2-} with distilled water, the samples were dried at 378 K in an oven for 30 min and milled in a mortar. Then the samples were calcined at 673 K for 1 h to prepare N- and S- doped TiO_2 (denoted as TNST) and then grinded for use. TiO_2 of Degussa P-25 was supplied by Degussa, and used as received without any pretreatment for comparison. TiO_2 and TNST were added into 0.5 mM solution of coumarin. The obtained samples were denoted as CODST and CONSDST, respectively. High Resolution Scanning electron micrograph (HRSEM) (Hitachi S-4800 Scanning Electron Microscope), ABrunauer Emmett Teller (BET) surface measurement device (Model ASAP 2100, Micromeritics, USA), Elemental Analyzer (EA) (Elementar VarioEL-III, German)and ultraviolet-visible (UV/vis) spectroscopy (UV/v is spectrophotometer, Evolvtion-220,Thermo., USA) were used for the characteristic analyses of these catalysts.

B. DSSC Preparation

TiO_2 and TNST were suspended in solutions of acetylacetone (2, 4-Pentanedione, NacalaiTesque Inc., Kyoto, Japan) and Triton X-100 (NacalaiTesque Inc., Kyoto, Japan). Then, the photocatalysts were uniformly coated on the indium-doping tin oxide (ITO) surface. The ITO film was calcined at 723 K for 20 min and the coated catalyst was 0.03 g. For the DSSC preparation, the ITO film was inserted into the solution of coumarin at 0.5 mM for 10 min. The ITO film, wetted with a film of the catalyst coating, was then driedat 378 K in an oven and denoted as negative electrode. The positive electrode was prepared by coating pure carbon film. The electrolyte was chosen as KI_3 solution. The DSSCs prepared from catalysts of TiO_2, TNST, CODST and CONSDST were denoted as TiO_2 solar cell (TSC), TNST solar cell (TNSSC), CODST solar cell (CODSSC) and CONSDST solar cell (CONSDSSC), respectively. The photoelectrochemical characteristics of DSSCs were analyzed by Cyclic Voltammetry (5600 Electrochemical Workstation, Jichan, Taiwan).

C. Light Source

In this work, the application of DSSC is focusing on the indoor circumstance or Building-integrated photovoltaic (BIPV). The light sources were chosen as visible light lamp (VLL) and lighting-emitting diode (LED) used in indoor lighting. The LEDis more novel, and has lower power, electromotive force (emf) demand and price, but higher efficiency, life-span and security as compared to a lamp. Also, it does not generate too muchheat. The visible light used in this study were red light emitting diode bar(RLED), blue light emitting diode bar (BLED), white light emitting diode bar(WLED) and visible light lamp(VLL) for the comparison light. Table I shows the characteristics of light sources used in this study.

TABLE I: CHARACTERISTICS OF THE LIGHT SOURCES USED IN THIS STUDY.

	Viewing angle	Power(W)	WL[e] range (nm)	Illumination (Lm[f])
RLED[a]	120°	2.64~2.88	625~630	25~30
BLED[b]	120°	2.04~2.4	465~470	24~24
WLED[c]	120°	2.04~2.4	300~700	60~70
VLL[d]	120°	8	440~760	90

a. Red light emitting diode bar. b. Blue light emitting diode bar.c. White light emitting diode bar. d. Visible light lamp. e. Wavelength. f. Luminous flux.

D. Apparatus

Four types of solar cells were used in this work: TSC, TNSSC, CODSSC and CONSDSSC. I–V characteristics were measured using the two-electrode system: WE and CE. A small resistance (50Ω) was used to convert current to voltage. The tested parameters for electricity and photoelectrochemical characteristics were open-circuited output voltage (V_{oc}), short-circuit output current (I_{sc}) and maximum power (P_m).A high input resistance X–Y recorder was used to record the I–V curves. The photosof I–Vcharacteristics were done by illuminating the solar cell using RLED, BLED, WLED and VLL, which were connected to a stabilized power supply. I–Vcurve of the sample was given in units of milli-Ampere (mA) or milli-Ampere per gram of catalyst (mA/g) vs. voltage (V). The intensity at the location of the sample was given in units of mW/cm^2.This was calculated from the readings of lux meter which gives the illumination at the surface of the sample in units of lux and theexact value of intensity was calculated using the illuminated area of the sample, knowing that the mechanical equivalent of light [25] is equal to 0.00161 W/lm and1 lux=1 lm/m^2.

III. RESULTS AND DISCUSSION

A. Characteristics of Dye-Sensitized N-and S-doped TiO_2

1) HRSEM

HRSEM were taken for TiO_2, $TiN_xO_{2-x}S_y$ and dye-sensitized samples, as illustrated in Fig. 1 From Fig. 1(a) and 1(b), the sizes of the TiO_2 and CODST were 40.98 and 44.26 nm, respectively. After the dye sensitization of TiO_2, the particles became larger than those without sensitization because of the aggregation phenomenon. The aggregated phenomena may have been due to the electrostatic attraction. From more high revolution image (Fig. 1(e)-1(f)), the sizes of TNST and CODST were between 15–25 nm. The particles of N- and S-doped TiO_2 with or without sensitization were all smaller and more uniform than those of TiO_2. It is believed that the flocculent settling in the precipitation process of $TiN_xO_{2-x}S_y$ let the nucleation mechanism appearing Ostwald ripening [22]. Therefore, the particle size was smaller than that of TiO_2. The HRSEM resolution was between 2–5 nm; therefore, the N particle could not be detected by HRSEM due to the limited resolution of the microscope [17].

2) UV/Vis absorption spectra

Fig. 2 shows the UV/Vis absorption spectra graph of coumarin. There are two major peaks of coumarin and the first and second absorption peaks are at 343 and457.5~541.5 nm, respectively. The two peaks are near the ranges of UVC and blue to green light, respectively. Therefore, the light source of long wavelength is fevered for coumarin.

(a)   (b)

(c)   (d)
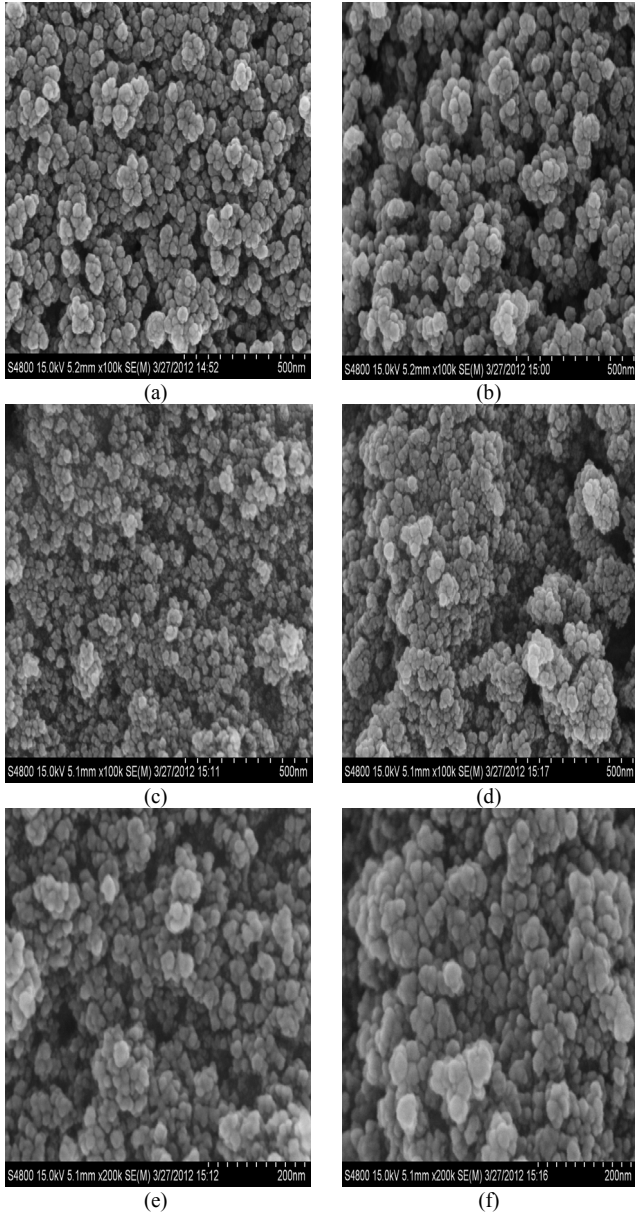
(e)   (f)

Fig. 1 Photos of SEM of (a)TiO2(100,000 ×), (b) CODST(100,000 ×), (c) TNST(100,000 ×), (d) CONSDST(100,000 ×), (e) TNST (200,000 ×), (f) CONSDST(200,000 ×).
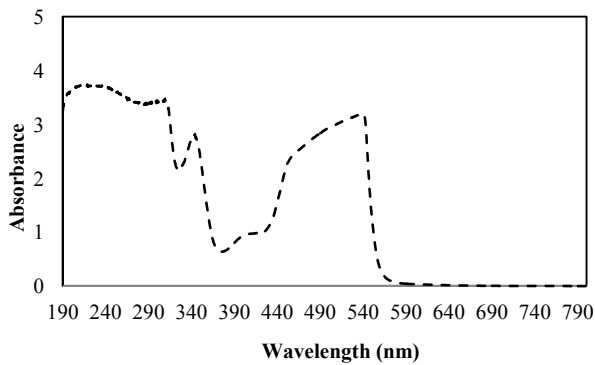

Fig. 2. UV/v is absorption spectra of coumarin solutions. Concentration of coumarin=0.5 mM.

3) Elemental analyses

The results of the elemental analyses for CODST, TNST and CONSDST are listed in Table II. The doping N source is derived from the ammonia and dye, and the mass percentage of N is in the order of CONSDST >TNST>CODST. The S source is contributed from Ti $(SO_4)_2$, and it is the highest

value among the analyzed elements. Also their order is the same as that of N component. Comparison with the sum of all elements, including N, C, S and H, CONSDST has the highest mass percentage, and the value is about 3.21 wt. %.

C and H element in CONSDST totally contain about 38.94 wt. % and the value is larger than that of TNST (20.56 wt. %), the reason may be due to that the adsorption of coumarin is pure hydrocarbon ($C_9H_6O_2$).

TABLE II: Elemental Analysis of Dye-sensitized Catalysts[a]

	N (wt. %)	C (wt. %)	S (wt. %)	H (wt. %)	Total
CODST	0.03	0.12	0.09	0.13	0.37
TNST	0.19	0.07	1.51	0.37	2.14
CONSDST	0.29	0.31	1.67	0.94	3.21

a. Accuracy: ±0.1 %. Precision: ±0.2 %.

4) BET surface area

The BET surface areas of TiO$_2$,CODST,TNST and CONSDST calculated by BET equation are listed in Table III. Using the precipitation method, the BET surface area of TNST increased to 120.71 m^2g^{-1}, and was larger than those of commercial TiO$_2$,CODST and CONSDST by about 2.33, 2.25 and 1.17 times, respectively. The reason of the larger BET surface area of TNST was believed to that TNST have smaller particle size (15～25 nm) [17].Dye-sensitization on the surface of TiO$_2$ and TNST changed the BET surface area, such as the case of CONSDST with the decrease of 17.79 m^2g^{-1}. The reason is believed to that dye concentrated o blocked into the pore of nanop articles. The adsorption average pore diameter and desorption average pore diameter are opposite against to the BET surface areas. Therefore, Dye-sensitization increased the adsorption average pore diameter and desorption average pore diameter.

TABLE III: Bet Surface Analysis and Pore Size of Dye-sensitized Tio2 andTnst

Sample	Area		Pore Size	
	BET surface area (m²/g)	Langmuir surface area (m²/g)	BJH Adsorption average pore diameter (4V/A) (Å)	BJH Desorption average pore diameter (4V/A) (Å)
TiO$_2$ [17]	51.85	-	-	-
CODST	53.64	75.28	310.62	292.32
TNST	120.71	168.50	114.86	107.78
CONSDST	102.92	141.52	194.91	183.48

B. Photoelectro chemical Characteristics of Dye-Sensitized N-and S-doped TiO$_2$

Fig. 3 shows I-V curves of all DSSCs at different light sources. From Fig. 3, the highest value of P$_{max}$ is 4.81 µW at VLL for CONSDSSC. Among all light irradiations, CONSDSSC have the highest P$_{max}$ and they are all in the order of VLL>WLED > BLED>RLED. After the dye sensitization of TNST, the increase of P$_{max}$ from TNSSC to CONSDSSC is clear, and it is about 2.15 to 3.76 times between the three light sources of BLED, RLED and WLED. It is evidenced that both N- and S-doping and dye sensitization are the key factors for DSSC to improve their efficiency. The TNST has smaller crystalline size and larger

specific surface area and can enhance the adsorption amount of coumarinthan that of commercial TiO_2. Surface oxygen defects produced by nitrogen and sulfur doping would improve the adsorption of coumarin and excited electronto transfer to the conduction band of TNST. Therefore the visible light activity of the sensitized N- and S-doped TiO_2 is much higherthan that of sensitized TiO_2 at different wavelength light of indoor LED.
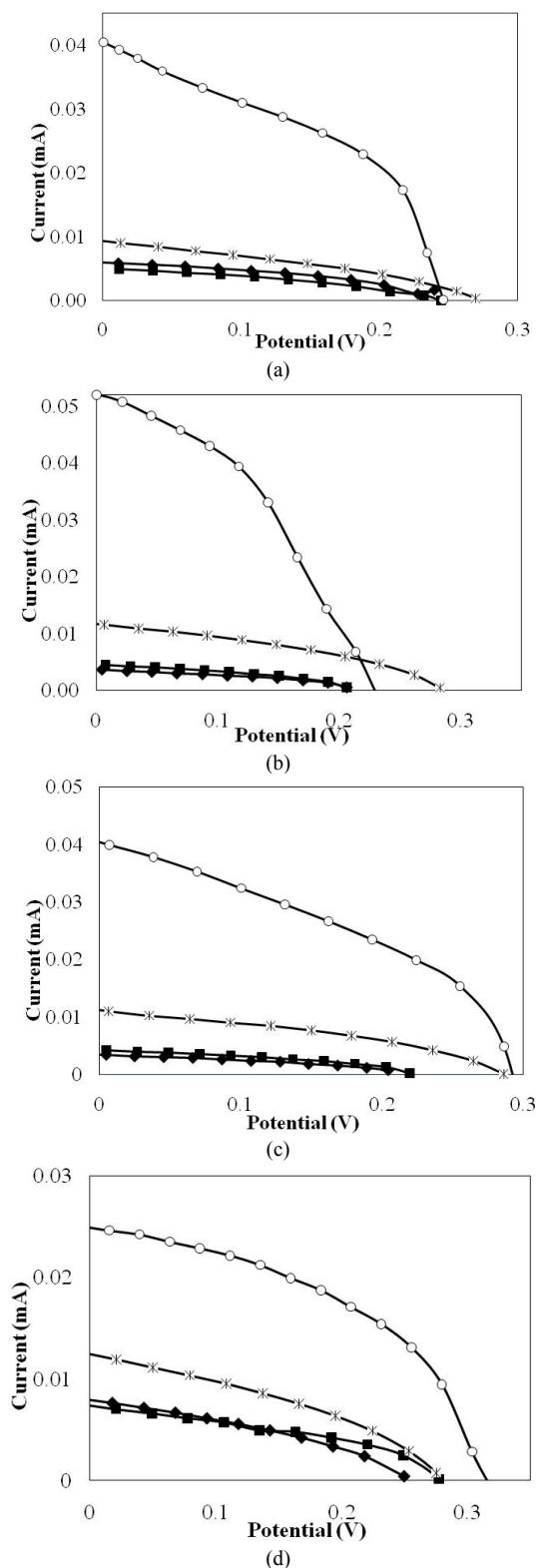

Fig. 3. I-V curves of different DSSCs at the irradiations of light bars.
○:CONSDSSC,＊: TNSSC,◆: TSC,▉: CODSSC.
Catalyst mass=0.02 g, coumarin=0.5 mM and 0.5 mL. (a) RLED, (b) BLED, (c) WLED, (d) VLL.

IV. CONCLUSION

The dye-sensitized N- and S- doped TiO_2 ($TiN_xO_{2-x}S_y$, TNST) with coumarin (CONSDST) with high visible activity was prepared and setup as DSSC, then tested in different visible lightsources. The sensitization of coumarinin TNST extends the visible light response range obviously. The TNST has smaller crystalline size and larger specific surface area and can enhance the adsorption amount of coumarin than that of commercial TiO_2.Surface oxygen defects produced by nitrogen and sulfur doping would improve the adsorption of coumarin and excites electronto transfer to the conduction band of TNST. Therefore the visible light activity of the sensitized TNST is much higher than that of the sensitized TiO_2 at different wavelength light of indoor LED.

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