Chemical Physics Letters 714 (2019) 6–10

Contents lists available at ScienceDirect

Chemical Physics Letters

journal homepage: www.elsevier.com/locate/cplett

Research paper

$CdSe/TiO_2$ nanotubes for enhanced photoelectrochemical activity under solar illumination: Influence of soaking time in CdSe bath solution

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HIGHLIGHTS

- $CdSe/TiO_2$ was prepared via chemical bath deposition chemosynthesis method.
- Optimum CdSe loaded on TiO_2 nanotubes played a crucial role in PEC response.
- 30 min soaking time for $CdSe/TiO_2$ nanotubes showed a maximum photocurrent density.
- CdSe dopants loaded on TiO_2 nanotubes act as efficient mediators for electrons.

ARTICLE INFO

Keywords:
$CdSe/TiO_2$ nanotubes arrays
Photoelectrochemical activity
Chemical bath deposition
Recombination losses
Charge carriers

ABSTRACT

In the present study, nanotubular structure of cadmium selenide/titanium dioxide ($CdSe/TiO_2$) was successfully prepared via chemical bath deposition (CBD) chemosynthesis method to improve photoelectrochemical activity under solar illumination. The results revealed that optimum CdSe dopants incorporated into TiO_2 nanotubes played a crucial role in determining the photoelectrode performance in water splitting. It was found that synthesized $CdSe/TiO_2$ nanotubes soaked at 30 min of CdSe precursor showed a maximum photocurrent density of 0.0016 A/cm^2 with improvement of about 10% than that of pure TiO_2 nanotubes under solar illumination. The presence of optimum contents of CdSe dopants incorporated into TiO_2 nanotubes could act as efficient mediators for trapping photo-induced electrons and reduce the recombination losses of charge carriers significantly.

1. Introduction

Recently, several efficient, and selective synthesis methods to fabricate active and efficient $CdSe/TiO_2$ photocatalyst have been explored to develop solar driven photocatalysis and photoelectrochemical fields. The physiochemical properties of TiO_2 photocatalyst can be tuned by incorporating an optimum amount of CdSe dopants to reduce recombination charge carrier losses. To date, several preparation methods can be applied to fabricate hybrid $CdSe/TiO_2$ photocatalyst either in particle, thin film, or nanostructured forms. Based on the literature, common preparation methods are vacuum evaporation [1,2] electrochemical deposition [3], sol-gel [4], spray pyrolysis [5], successive ionic layer adsorption and reaction method (SILAR) [6], radio-frequency sputtering [7,8], chemical vapour deposition [9], as well as chemical bath deposition (CBD) technique [10–12]. Among various preparation methods available, CBD technique has been appeared as most promising technique to form $CdSe/TiO_2$ photocatalyst due to the economic and simple procedures without any sophisticated instruments. Indeed, tunable optical and band gap energy for TiO_2 nanotube arrays' can be achieved via CBD chemosynthesis method. It's facile and cost-effective method with additional advantages such as reproducibility, minimum material wastage, no need of handling poisonous gases, no requirement of sophisticated instruments, and well suitable for economical way of large area deposition at relatively low temperatures [13,14]. In fact, working principle of CBD chemosynthesis method is based on the principle of ion-by-ion condensation in a solution state like the atom-by-atom deposition in a vacuum process [15].

In general, CBD chemosynthesis method has been applied extensively to fabricate chalcogenide CdSe thin films for window coated layer material in solar powered panel fabrication [16]. Herein, detailed studies of CdSe dopants incorporating into TiO_2 nanotubes through CBD chemosynthesis method by optimizing the growth parameter of soaking time have been carried out. The obvious change in crystal structures of $CdSe/TiO_2$ photocatalyst was observed, such as

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https://doi.org/10.1016/j.cplett.2018.10.054
Received 21 September 2018; Received in revised form 18 October 2018; Accepted 23 October 2018
Available online 23 October 2018

amorphous, cubic, as well as mixture of cubic and hexagonal phases [14,17,18]. Robel et al. reported on the use of bifunctional surface modifiers (SH-R-COOH) to sensitize CdSe quantum dots onto mesoscopic TiO_2 films, which were employed as a photoanode in a photoelectrochemical cell [19]. Besides, Zhang et al. demonstrated the introduction of CdSe dopants by CBD into the TiO_2 nanotubes to enhance the photocathodic protection of stainless steel [20]. The basic principle underlying the deposition of CdSe dopants is based on the slow release of Cd^{2+} and Se^{2+} ions from its complex in a basic aqueous bath. Subsequently, it condenses through an ion-by-ion basis onto the TiO_2 nanotubes substrates that are vertically mounted in the reaction bath [13]. Dissociation of Cd^{2+} and Se^{2-} ions from its complex either allow the CdSe nanocrystals to incorporate gradually on TiO_2 nanotubes or aggregate into larger undesired particles, while it can be manipulated by controlling the CBD chemosynthesis's soaking time. Theoretically, low concentration of Cd^{2+} and Se^{2-} ions is sufficient to yield the solid phase CdSe nanocrystals and eventually form hybrid CdSe/TiO_2 photocatalyst [14,16].

Basically, source of cadmium can be prepared from its salts or oxide to form a complex by slowly adding alkaline (i.e. ammonium solution) under agitation until a clear solution was obtained. The steps of formation of precursor of Cd complex ions are as below (Eqs. (1)–(3)):

$$Cd(CH_3COO)_2 + 2NH_4OH \rightarrow Cd(OH)_2 + 2NH_4(CH_3COO) \quad (1)$$

$$Cd(OH)_2 + 4NH_4OH \leftrightarrow Cd(NH_3)_4{}^{2+} + 2(OH^-) + 4H_2O \quad (2)$$

$$Cd(NH_3)_2{}^{4+} \leftrightarrow Cd^{2+} + 4NH_3 \quad (3)$$

Meanwhile, freshly prepared sodium selenosulphate (Na_2SeSO_3) solution acts as the source of Se^{2+} ions. It can be prepared through heating of Se metal or its oxide with sodium sulphite under reflux. The reduction of Na_2SeSO_3 to elemental Se can be described through the following reaction (Eq. (4)) in the aqueous ammoniacal medium.

$$Na_2SeSO_3 + 2OH^- \rightarrow Na_2SO_4 + H_2O + Se^{2+} \quad (4)$$

Hence, the overall chemical reaction is presented as below (Eq. (5)):

$$Cd(NH_3)_2{}^{4+} + Na_2SeSO_3 + 2OH^- \rightarrow CdSe + 2NH_3 + Na_2SO_4 + H_2O \quad (5)$$

To the best of our knowledge, detailed studies on CdSe dopants incorporated TiO_2 nanotubes via CBD chemosynthesis method and their photoelectrochemical activity under solar illumination are still lacking. In the present study, CBD chemosynthesis method was applied to prepare hybrid CdSe/TiO_2 nanotubes to overcome the main drawback of recombination losses of charge carriers by forming lattice electron traps. For nanotubes structure, capillary action of the tube draws the CdSe-containing solution into the pores. Then, the small CdSe dopants in the solution could diffuse slowly into the bulk TiO_2. A minimum of 30 min soaking time is required for completion diffusion process. Thus, the controlled soaking time for the synthesis of CdSe/TiO_2 nanotubes is a crucial step in improvement of photoelectrochemical activity. This study aims to identify the optimum soaking time of CBD chemosynthesis method to obtain the desired hybrid CdSe/TiO_2 nanotubes for the best solar-driven photoelectrochemical water splitting activity.

2. Experimental procedures

Well-aligned TiO_2 nanotubes were produced via electrochemical anodization of titanium (Ti) foil at 40 V in a bath with ethylene glycol electrolyte composed of 0.3 wt% of ammonium fluoride (NH_4F), and 5 wt% of hydrogen peroxide (H_2O_2) for 60 min. The as-anodized TiO_2 nanotubes film was then dried in an ambient condition. All anodized samples were further annealed at 400 °C for 4 h in air atmosphere and the heating rate was fixed at 5 °C/min and naturally cool down to obtain desired anatase phase of TiO_2 [21,22]. The most important stage was formation of CdSe/TiO_2 nanotubes through chemical bath deposition technique. Herein, precursor of selenide (Na_2SeSO_3 solution) was prepared via mixture of 0.6 M of sodium sulphite (Na_2SO_3) and 0.2 M of selenium metal powder in 100 ml distilled water then heated under reflux at 90 °C for 3 h to form a clear Na_2SeSO_3 solution. Meanwhile, cadmium precursor was formed by dissolving 0.2 M cadmium acetate dehydrate $Cd(CH_3COO)_2{\cdot}2H_2O$ in 50 ml deionized water. Next, a concentrated ammonia solution (30%) was added into mixture slowly. Ammonia was used to adjust the pH of the solution between 12 and 12.5 to prevent the reverse reaction of $Cd(NH_3)_4{}^{2+}$ to form stable cadmium hydroxide $Cd(OH)_2$. The soaking time of CBD technique in CdSe bath solution for well-aligned TiO_2 nanotubes were varied from 0.5 h to 10 h.

The hybrid CdSe/TiO_2 nanotubes' surface and cross-sectional morphologies was studied via Field Emission Scanning Electron Microscopy (FESEM) using a JEOL JSM 7600-F operated at 5 kV and 10 kV with working distance of approximately 1 mm, whereas the element analysis was performed via Energy Dispersive X-ray Spectroscopy (EDX) analysis with an Oxford Instruments. Moreover, samples' phase transition was determined via both Raman Spectroscopy and X-ray diffraction (XRD). Raman spectrometer (Renishaw inVia, United Kingdom) was operated over the range of 100–1000 cm^{-1}, at an excitation wavelength of 532 nm generated by an Ar ion laser. XRD (Bruker AXS D8 Advance, Germany) analysis was conducted using a Cu target (Nickel filter), Kα radiation (λ = 0.1546 nm) at scanning rate over angle, 2θ in the range 20–70°. The photoelectrochemical activities of hybrid CdSe/TiO_2 nanotubes were further characterized using a three electrodes water splitting cell under solar irradiation. The working electrode, platinum rod was served as the counter electrode whereas Ag/AgCl in saturated KCl was served as the reference electrode. The used electrolyte was composed of 1 M potassium hydroxide aqueous solution. All electrodes within water splitting cell were connected to the potentiostat (Autolab PGSTAT 204, Netherlands) by using a control software NOVA for linear sweep potential as photoelectrochemical measurements. The current range is fixed from 10 mA to 10 nA. A 150 W Xenon lamp (Zolix LSP-X150, China) was used as the light source, with intensity 100 mW/cm^2 light beam was focused on the immersed portion of working electrode.

3. Results and discussion

Based on our previous studies, well-aligned TiO_2 nanotubes was successfully fabricated via electrochemical anodization of Ti foil in ethylene glycol electrolyte composed of 0.3 wt% NH_4F and 5 wt% H_2O_2 at 40 V for 60 min then subsequently annealed at 400 °C to form anatase phase of TiO_2 nanotubes film. Hence, in the following studies, TiO_2 nanotubes film was synthesized in above-mentioned conditions to investigate the influence of soaking time of CBD technique on the formation of CdSe/TiO_2 nanotubes. It's a well-known fact that controlling soaking time of CBD technique is crucial in determining the generation rate of the Cd complex and Se anions as well as combination of Cd complex and Se anions into clusters. Thus, these CdSe clusters might be aggregated into desired nano-sized particles under an optimum soaking time of CBD. Therefore, optimization of the soaking time during the CBD process for CdSe/TiO_2 nanotubes film is crucial in improving the photoelectrochemical activity under solar illumination.

Herein, deposition of the CdSe dopants into TiO_2 nanotubes was conducted in a bath solution containing of both Se^{2-} and Cd^{2+} precursor. It's a well-known fact that once the ionic product of Cd^{2+} and Se^{2-} ions were exceeded solubility product of CdSe (i.e., IP ≥ SP (10^{-33})), sufficient CdSe dopants deposition took place on the TiO_2 nanotubes as stated in the following steps:

Sodium selenosulphate (Na_2SeSO_3) hydrolyzes in solution to give Se^{2-} ions as presented in Eqs. (6) and (7):-

$$Na_2SeSO_3 + OH^- \rightarrow Na_2SO_4 + HSe^- \quad (6)$$

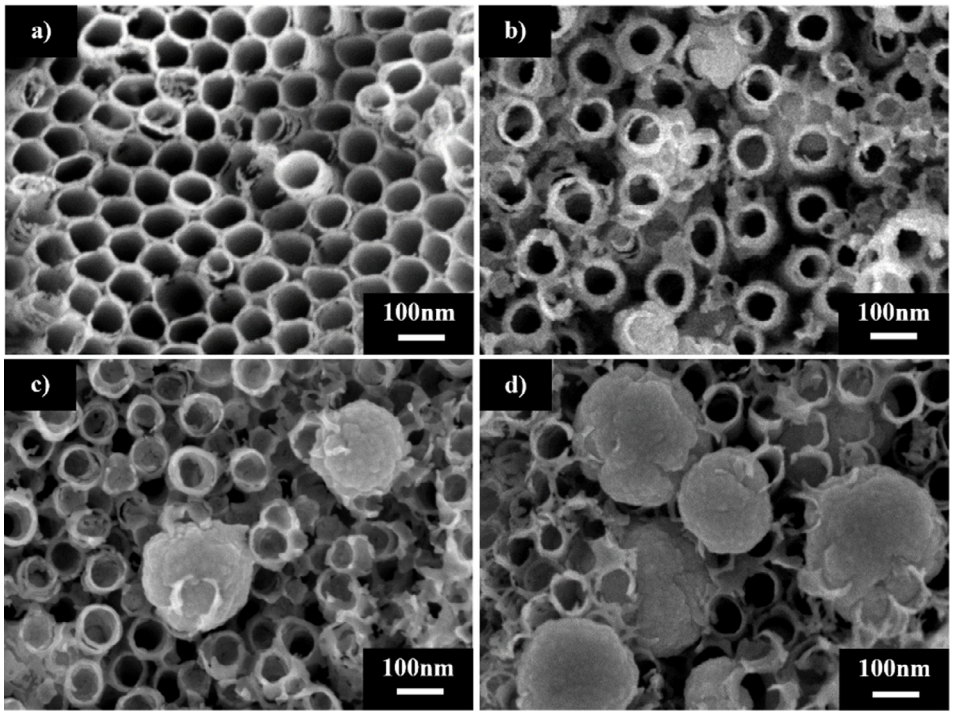

Fig. 1. FESEM images of CdSe/TiO_2 nanotubes soaked for (a) 30 min, (b) 3, (c) 5, and (d) 10 h in 5 mM CdSe solution.

$$HSe^- + OH^- \rightarrow H_2O + Se^{2-} \quad (7)$$

Ammonia solution was added in Cd salt solution to form the complex cadmium tetraammine ion [$Cd(NH_3)_4{}^{2+}$] as showed in Eq. (8):-

$$Cd^{2+} + 4NH_3 \rightarrow Cd(NH_3)_4{}^{2+} \quad (8)$$

Then $Cd(NH_3)_4{}^{2+}$ reacts with hydrolyzed Se^{2-} ion to form CdSe as described in Eq. (9):-

$$Cd(NH_3)_4{}^{2+} + Se^{2-} \rightarrow CdSe + 4NH_3 \quad (9)$$

FESEM micrograph in Fig. 1 represents the influence of soaking time of CBD from 30 min to 10 h towards the annealed pure TiO_2 nanotubes. Fig. 1(a) shows that the top view of the nanotubes arrays after soaking in a bath solution containing of both Se^{2-} and Cd^{2+} precursor for 30 min. Interestingly, it could be noticed that the morphological appearance view is about the same as pure TiO_2 nanotubes with opening tubes without any aggregated CdSe nanoparticles or nanoclusters on their surface. Generally, the formation of CdSe precipitate from a solution involves the steps of nucleation and particle growth. Both processes take place between the reactions of slowly released metal ions and chalcogen ions in aqueous solution. There is some minimum number of both ions required to produce a stable phase called as nucleus. Nucleus form in solution will either undergo decomposition reverse to ionic form or in contact of particles or combine to grow up to a certain size of precipitate. This is called the concept of nucleation in solution. In the time of 30 min, it was insufficient for the nucleation process to form significant precipitate. An average content of both Cd and Se for 30 min soaking duration was about 0.46 at% and 0.39 at%, respectively based on EDX analysis. At the soaking duration of 3 h [Fig. 1(b)], CdSe nanoclusters formed and surrounded the pores entrance and thus narrowing the pore entrance nanotubes' diameter to 50 nm. Besides, some CdSe spheres were clogged on the pore entrance were observed and content of Cd and Se were approximately 2.85 at% and 2.69 at%, respectively. Further increased in soaking duration to 5 and 10 h, most of the nanotubes were covered by the precipitate and CdSe spheres with the size of 230 nm and 300 nm, respectively. The EDX data collection showed both the content of Cd and Se dopants were increasing with the soaking duration in CBD process. At 5 h of soaking duration, an average content of Cd and Se dopants were 5.04 at% and 4.82 at%, respectively. For 10 h soaking duration, an average elemental composition of Cd and Se dopants were 8.89 and 8.25 at% respectively. All of the obtained results manifested that soaking time of CBD process played a crucial role in determining the surface morphologies of CdSe/TiO_2 nanotubes film. An average elemental composition for titanium, oxygen, cadmium and selenide within pure TiO_2 and CdSe/TiO_2 nanotubes at different soaking time based on the EDX spectra are summarized in Table 1.

Next, XRD analyses were carried out to identify the crystal plane and phase of hybrid CdSe/TiO_2 nanotubes soaked for different durations in 5 mM Cd and Se precursor solution. The XRD spectra were shown in Fig. 2. It shows similar result as our previous studies. Intense peaks from the XRD pattern at 25.37°, 38.67°, 48.21°, 54.10°, 55.26°, 62.66° and 68.74° are corresponding to (1 0 1), (1 1 2), (2 0 0), (1 0 5), (2 1 1), (2 0 4) and (1 1 6) crystal planes of anatase phase of TiO_2 [JCPDS No 21-1272]. Ti substrate found with the diffraction peaks at 35.1°,38.4°,40.2° and 53.0°, which correspond to the crystal planes of (1 0 0), (0 0 2), (1 0 1) and (1 0 2) respectively [JCPDS No 44-1294]. The highest peak is contributed by overlapping the peak of anatase crystal plane (1 0 1) and (1 1 1) plane of cubic CdSe [JCPDS No 19-0191]. Interestingly, it could be noticed that peak intensity was increasing with increased of soaking duration from 3 h to 10 h at 25° of XRD spectra, which indicating that increased in crystallinity at the crystal plane (1 1 1) of cubic CdSe. Besides, a small characteristic diffraction peak of (3 1 1) planes of cubic zinc blended phase CdSe at 49.7°

Table 1
An average at% of CdSe/TiO_2 nanotubes soaked in 5 mM CdSe solution at different soaking times obtained by EDX analysis.

Soaking time (h)	Atomic percentage (at%)			
	Ti	O	Cd	Se
0.5	37.92	61.23	0.46	0.39
3	37.98	56.48	2.85	2.69
5	34.18	55.96	5.04	4.82
10	32.79	50.07	8.89	8.25

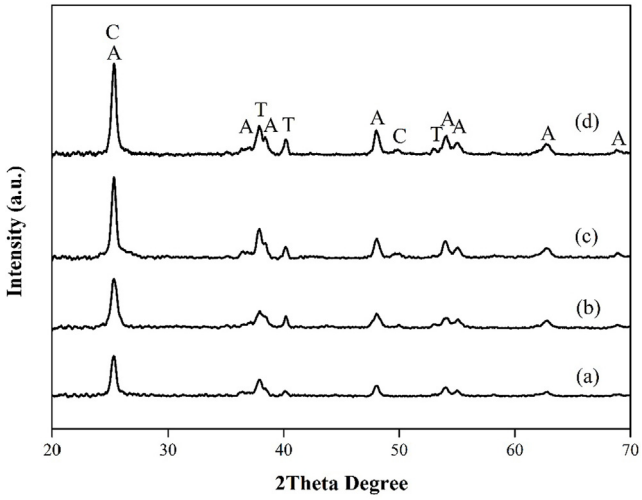

Fig. 2. XRD diffraction patterns of CdSe/TiO_2 nanotubes soaked for (a) 30 min, (b) 3, (c) 5 and (d) 10 h in 5 mM CdSe solution [A = anatase, T = Ti metal, C = CdSe].

was appeared in the XRD spectra for samples applied 3, 5 and 10 h of soaking duration. The peak intensity was more obvious with the soaking duration, and indicates the abundancy of the CdSe crystal plane equivalent as the EDX analysis. From the XRD pattern, the sample soaked for 30 min shows no obvious cubic zinc-blended CdSe peaks due to lower content of CdSe (< 2 at% from EDX analysis). This is due to little amount of CdSe which gives insufficient response to detect by XRD analysis [23,24].

Next, Raman analysis clearly to show four prominent bands at 144, 394, 515 and 636 cm^{-1} were attributed to the E_g, B_{1g}, A_{1g}, and E_g vibration modes of anatase phase TiO_2 respectively as shown in Fig. 3. [25–27]. A gradual decreased in the intensity of the four anatase bands was observed with increased deposition duration. It was due to the increase of the CdSe content as shown in EDX analysis (Table 1) as solid phase precipitate covered on the surface of TiO_2 nanotubes substrate. Hence, this gradually reduces the signal received in the Raman analysis. Furthermore, a Raman band at 206 cm^{-1} corresponds to the first order cubic CdSe longitudinal optical phonon (LO) was observed at the Raman spectra of the deposition duration 3, 5 and 10 h. This further confirmed the presence of cubic CdSe phase in XRD measurement with the trend shown by EDX elemental analysis discussed above.

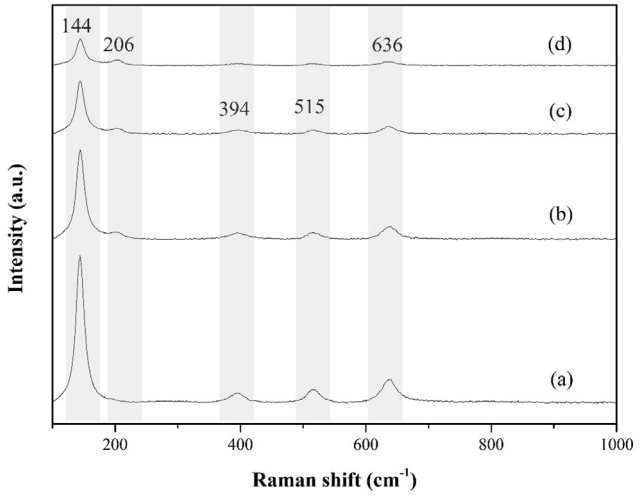

Fig. 3. Raman spectrum of CdSe loaded TiO_2 nanotubes soaked for (a) 30 min, (b) 3, (c) 5, and (d) 10 h in 5 mM CdSe solution.

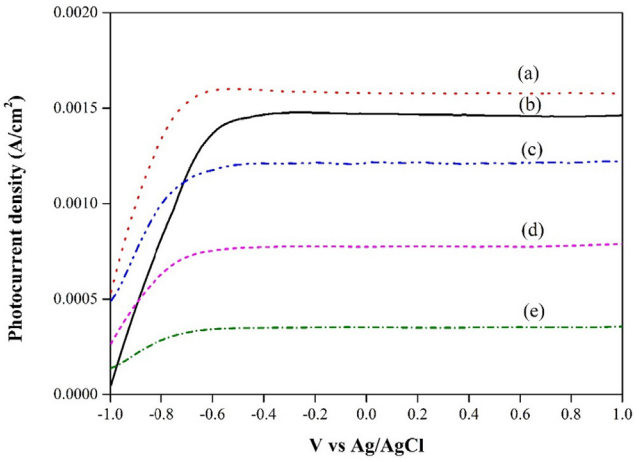

Fig. 4. The j_p-V characteristic curves of CdSe-TiO_2 nanotubes soaked at different duration of CdSe precursor: (a) 30 min (red dotted line), (b) pure TiO_2 nanotubes (black straight line), (c) 3 h (blue dotted line), (d) 5 h (pink dotted line), and (e) 10 h (green dotted line). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

A photocurrent density–potential characteristic plot for the different deposition durations of the hybrid CdSe/TiO_2 nanotubes in 5 mM CdSe precursor is shown in Fig. 4. A maximum photocurrent density of 0.00160 A/cm^2 with improvement of about 10% was noticed for CdSe/TiO_2 nanotubes subjected to 30 min soaking time in precursor solution, which was slightly higher as compared to pure TiO_2 nanotubes (0.00145 A/cm^2). Nevertheless, hybrid CdSe-TiO_2 nanotubes subjected to 3, 5, and 10 h soaking times exhibited decreased trend in photocurrent density–potential characteristic with 0.00120, 0.0080 and 0.0035 A/cm^2, respectively. The controlled soaking time in CdSe precursor solution for the synthesis of CdSe/TiO_2 nanotubes is a crucial step in the improvement of photocurrent density–potential characteristic. A small amount of Cd and Se content (< 0.50 at%) within TiO_2 nanotubes showed better photocurrent density–potential characteristic than that of pure TiO_2 nanotubes. This finding may be attributed to the low contents of CdSe species underwent the diffusion within the TiO_2 lattice. In this manner, optimum content of CdSe species acted as an effective electron acceptor, which generates an energy band level below the conduction band of TiO_2 itself; thus, CdSe species could trap or grab the photogenerated electrons from TiO_2 due to the interpretation of inter-band states position with key redox potential or surface states within a corresponding energetic position. Nevertheless, excessive loading amount of CdSe species on TiO_2 nanotubes exhibited poor photocurrent density–potential characteristic. These results inferred that diffusion of CdSe species into the lattice of TiO_2 reached a saturation status and started to cover by the precipitate and CdSe spheres, which acted as defect sites or recombination sites for the charge carriers. This condition hindered the transportation of electrons to the Ti substrate and led to poor photocurrent density–potential characteristic [28–31]. Thus, the improvement in photocurrent density–potential characteristic under solar illumination is ascribed to the optimum content of CdSe species incorporated into the lattice of TiO_2.

4. Conclusion

In summary, considerable effort has been conducted to further improve the photocurrent density–potential characteristic under solar illumination by loading optimum content of CdSe species on TiO_2 nanotubes using the CBD chemosynthesis method. CdSe/TiO_2 nanotubes subjected to 30 min soaking time in precursor solution exhibited high photocurrent density–potential characteristic than that of other samples. An obvious improvement in the photocurrent density–potential

characteristic could be observed when the loading at% for CdSe species on TiO_2 nanotubes was less than 0.5 at%, which could be effective electron trappers to minimize the recombination losses during solar illumination. Nevertheless, excessive loading at% for CdSe species on TiO2 nanotubes could reach a saturation level and negative effects could be happen with the formation of CdSe spheres on the opening mouth of nanotube arrays, which might be attributed to the high number of recombination spots for charge carriers.

Acknowledgements

This research work was financially supported by the University Malaya Research Grant (No. RP045B-17AET), University Malaya Research Fund Assistance (BKP) Grant (No. BK096-2016), and Global Collaborative Programme – SATU Joint Research Scheme (No. ST008-2017).

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