Retraction

Retraction: N719 Sensitized Solar Cell Features of Photoanodes Prepared with CDS Coated Hydrothermally Derived Anatase TiO2 Nanobelts (J. Phys.: Conf. Ser. 1916 012231)

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This article (and all articles in the proceedings volume relating to the same conference) has been retracted by IOP Publishing following an extensive investigation in line with the COPE guidelines. This investigation has uncovered evidence of systematic manipulation of the publication process and considerable citation manipulation.

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IOP Publishing regrets that our usual quality checks did not identify these issues before publication, and have since put additional measures in place to try to prevent these issues from reoccurring. IOP Publishing wishes to credit anonymous whistleblowers and the Problematic Paper Screener [1] for bringing some of the above issues to our attention, prompting us to investigate further.

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N719 Sensitized Solar Cell Features of Photoanodes Prepared with CDS Coated Hydrothermally Derived Anatase TiO$_2$ Nanobelts

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Abstract. Herein, DSSC performance of the photo-anodes of (i) TiO$_2$ powders derived from simple basic precipitation (SP) reaction (SP-TiO$_2$-1), (ii) TiO$_2$ powders derived from conventional hydrothermal reaction (CH-TiO$_2$-2) and (iii) hydrothermally derived TiO$_2$ coated with Cds by dip method (Cds-CH-TiO$_2$-3) is reported. The XRD, UV-visible spectroscopy, FESEM and HRTEM techniques are used for the characterization of resultant films. The characterization studies indicated the crystallization of anatase TiO$_2$ in all resultant films and Cds with hexagonal symmetry in case of Cds-CH-TiO$_2$-3 film. The crystallization of spherical agglomerates having average size of 36.05 nm with nearly uniform size distribution is realized in case of SP-TiO$_2$-1 by using FESEM. Further, HRTEM indicated the coating of Cds nanoparticles with average particle size of 15.86 nm on hydrothermally crystallized anatase TiO$_2$ nanobelts of width 200 nm in case of Cds-CH-TiO$_2$-3 films. The N719 sensitized solar cell properties these photoanodes obtained at 1000 W/m$^2$ input power indicated the enhancement of photoconversion efficiency ($\eta$) by 95.83 % in case of CH-TiO$_2$-2 ($\eta = 1.41$) as compared to SP-TiO$_2$-1 ($\eta = 0.72$) photoanode whereas 34.75 % in case of Cds-CH-TiO$_2$-3 ($\eta = 1.90$) as compared to CH-TiO$_2$-2 photoanode. The improvement in photoconversion efficiency ($\eta$) in case of CH-TiO$_2$-2 and Cds-CH-TiO$_2$-3 can be attributed due to increase in charge transfer current density ($J$) and decrease in re-combination rate of electron at electrode-electrolyte/dye contact. This is due to blocking of back scattered electrons and effective
electron-hole charge separation created by co-sensitizer CdS nanoparticles coated on TiO₂ nanobelts.

**Keywords:** TiO₂; CdS; Co-sensitizer; DSSC; Photoconversion efficiency.

1. Introduction
The solar cells sensitized with dye (DSSCs) have been considered as the potential devices for the sustainable energy supply due to their high environmental friendliness, low production cost and simple fabrication technology [1-3]. It is a technology alternate to other photovoltaic systems based on thin films of cadmium telluride, copper indium gallium diselenide, amorphous silicon etc. having moderate optical energy band gap [4-6]. Typical DSSC consist of three main components: semiconductor material photoanode film sensitized with dye, electrolyte of redox couple and counter electrode coated with thin metallic layer [7-8]. Generally, the fluorine or indium doped tin oxide (FTO or ITO) is used as substrate for the photoanode and counter electrode.

During last few decades, the wide band gap semiconductors have been studied rigorously due to their large number of applications. The photoanodes fabricated by using different nanostructures, nanocomposites and doped nanostructures and sensitized with various dyes have been employed for DSSC applications [9]. In these materials, nanocrystalline TiO₂ is noted to be most important material for large number of practical applications: energy conversion, sensing, optics, ceramic membrane, catalyst for photocatalytic decomposition etc. owing to its chemically inertness, biocompatibility, good stability against oxidation, nontoxicity and thermal stability [10-11]. The TiO₂ is used as photoanode material owing to better photo-conversion efficiency, low production cost and long-term photo-stability [12-13]. However, in case of DSSC made with TiO₂ photoanode, the photo-conversion efficiency is limited due to recombination of electrons injected into TiO₂ conduction band with the dye’s excited state at photoanode-dye/electrolyte interface. These back scattered electrons reduce the current density leading to decrease in photo-conversion efficiency. This recombination leads the regeneration of dye and/or electrolyte. Further, because of wide band gap of TiO₂ (~ 3.2 eV), the absorption of light is limited to ultraviolet region of solar spectrum [14]. The TiO₂ is having low electron mobility and it is also responsible for lower photo-conversion efficiency of DSSC [15]. Hence, for enhancing the photo-conversion efficiency of DSSC made with TiO₂ photoanode, different methods are adopted. TiO₂ nanostructures are found to be useful to create pathways thereby enhancing the electron mobility required for efficient photo-conversion efficiency phenomenon [16]. The energy efficient sensitizers like quantum dots, plasmonic nanoparticles, inorganic-organic dyes have been tried to increase the phenomenon of light harvesting in the visible region and hence for the photoconversion efficiency [17-18]. The different standard sensitizing dyes along with the cheap dye are reported for DSSC application [19-20]. For enhancing photo-conversion efficiency, TiO₂ photoanodes have been modified with various narrow band gap semiconductors or their quantum dots (QDs) due to their inherent nature of absorbing the light on visible region and ability to inject electrons to the TiO₂ conduction band [21-22]. The generation of additional-multiple electron-hole pairs and rapid charge separation due to high extinction coefficient and intrinsic dipole moments are main features of semiconductor QDs. It can help to improve photoconversion efficiency of DSSC to great extent [23-24]. In view of this, an attempt is made in present study to enhance efficiency of DSSC fabricated with TiO₂ photoanode. The effect of nanstructured morphology of TiO₂ derived by using conventional hydrothermal processing and TiO₂ photoanode co-sensitized with N719 dye and CdS nanoparticles on photoconversion efficiency of DSSC based on TiO₂ photoanode is studied in present work. The results obtained pertaining to this are analyzed and presented in current communication.

2. Methodology

*Preparation of nanocrystalline TiO₂ powders*
In one of the methods of preparation, the simple basic precipitation route as given in our earlier report was used for the synthesis of nanocrystalline TiO$_2$ powders [25]. The powders obtained by using this route at 300 and 450 °C were identified as SP-TiO$_2$-0 and SP-TiO$_2$-1 respectively. The SP-TiO$_2$-0 powder was used for conventional hydrothermal processing of nanocrystalline TiO$_2$ powder. Initially, NaOH solution of 10 M was made in double distilled water (DDW). The 1.1 g powder of SP-TiO$_2$-0 was mixed in 50 ml of aqueous NaOH solution. The conventional hydrothermal treatment was given to this mixture solution in a Teflon lined stainless steel vessel at 190 °C/24 h. The powder obtained in hydrothermal treatment was washed intensively with 0.1 N HCl and then with DDW for several times. The as-washed powder was heated at 100 °C/4 h. This whole procedure was carried out to remove the traces corresponding to the sodium titanate hydroxide [26] that was crystallized along with formation of titanium hydroxide as major phase. Finally, powder was heated at 400 °C/2 h in air atmosphere to obtain the well-crystalline nanostructured TiO$_2$ powder. The resultant powder obtained by using conventional hydrothermal processing was identified as CH-TiO$_2$-2.

Substrate preparation

Initially, fluorine doped tin oxide (FTO) substrates were cleaned by using detergent and DDW. After this, substrates were cleaned ultrasonically for 15 min. in AR grade acetone and then rinsed in acetone. Finally, under IR lamp the substrates were dried. On this thoroughly cleaned FTO substrate, TiO$_2$ blocking layer was deposited by using spin coating method. Finally, substrates were used instantaneously for the deposition of different photoanode films for DSSC application.

Preparation of photoanode films for DSSC application

The photoanode films of different powders were prepared by using Doctor Blade method on FTO substrates. 0.5 g of given powder was pulverized by using pestle-mortar and then mixed with 0.2 g of ethyl cellulose in ethanol to obtain the paste. To get lump free paste, few drops of acetyl acetone & 1 ml of α-terpineol was added and then excess ethanol was added to make paste suitably viscous. Then it was stirred at 250 rpm magnetically for 2 h. After magnetic stirring, the paste was sonicated for 2 h and finally used for deposition of photoanode film on substrate. For film deposition, scotch tape was used for masking all edges of substrate by leaving 0.15 cm$^2$ area for film deposition. In 0.15 cm$^2$ area, the paste of given powder was deposited. The as-deposited film was heated at 60 °C for removing the pinholes and finally film was annealed at 450 °C for 1 h. By using this procedure, the photoanode films of SP-TiO$_2$-1 and CH-TiO$_2$-2 powders were obtained and resultant films were identified as SP-TiO$_2$-1 and CH-TiO$_2$-2 respectively.

Preparation of CdS coated TiO$_2$ films

Initially, cadmium sulphate solution of 0.03 M and thiourea solution of 0.04 M were prepared in DDW. The 40 ml cadmium solution was heated at 50 °C and pH was maintained at ~ 9.0 by adding ammonia drop-wise. To this solution, the 40 ml solution of thiourea was added. The temperature was kept constant at 70 °C for resultant mixture solution. The colour of resultant solution was found to be turned into yellow. To this solution, the CH-TiO$_2$-2 photoanode film was dipped for 20 min. The CdS was uniformly deposited on CH-TiO$_2$-2 film and resultant CdS coated film was identified as CdS-CH-TiO$_2$-3.

Characterization of powders and films

The X-ray diffraction XRD patterns useful for phase analysis of as-prepared powders / films were recorded by using Rigaku X-ray diffractometer (MiniFlex 600 System, Japan, with λ = 1.5406 Å for CuK$_\alpha$ radiation). The absorbance spectra of as-prepared powders / films were recorded by using UV-visible spectrophotometer (UV - 2600 Shimadzu, Japan) to find band gap energy. For morphological analysis of as-prepared powders / films, the microphotographs were taken by using field emission scanning electron microscope: FESEM (Nova NanoSEM 450, EDS, Bruker XFlash 6i30) and high resolution field emission gun-transmission electron microscope: HRTEM (200 kV, JEOL JEM 2100F).

Fabrication of DSSC assembly and measurement of DSSC properties
The DSSC assemblies were made by using photoanodes of SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films. Initially, photoanode film was kept in N719 dye solution (0.5 mM) in ethanol for 24 h. The white colour of photoanode was noted to be changed to pink yellow due the absorption / loading of dye in the material of photoanode film. The dye sensitized photoanode film was assembled with platinum (Pt) coated FTO counter electrode with Pt side touching to the sensitized film. The electrolyte of requisite amount (0.05 M of iodine, 0.5 M of LiI, 0.5 M of 4-tertbutylpyridine 3-methoxypropionitrile) was poured between counter electrode and photoanode. Due to capillary action, the electrolyte solution was absorbed into DSSC cell. The proper DSSC assembly sealing was done. The positive and negative terminals of solar simulator system (Newport Corp., Oriel®, Sol2A®, Class ABA) were connected respectively to the photoanode film and counter electrode. The (J) current density - (V) voltage curves of resultant DSSCs were recorded at 1000 W/m² input power of the light incident from Neon lamp. The solar cell properties: V_{OC} (open circuit voltage), I_{SC} (short circuit current), FF (fill factor) and \eta (efficiency) were obtained from J-V curves and data recorded by the solar simulator system.

3. Result and Discussion

X-ray diffraction (XRD) studies

Figure 1 gives the X-ray diffraction: XRD patterns for SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films for 20 values between 20 to 80°. The well polycrystalline nature of SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films is realized from the observed intensities of different peaks in all XRD patterns. In all XRD patterns, the peaks for (101), (004), (200), (105), (211), (204), (116), (220) and (215) reflections corresponding to the anatase phase of TiO2 (JCPDS Card no. 21-1272) with tetragonal symmetry are observed. These peaks are marked with symbol ‘A’. The peaks matching to the rutile phase of TiO2 (JCPDS Card no. 21-1276) are not observed in all X-ray diffraction patterns. All films are thick due to the use of Doctor Blade method for their preparation. Hence, no peaks corresponding to FTO substrate used for preparation of films are found in these XRD patterns. Consequently, all XRD patterns of three films are indexed to the anatase TiO2 phase with tetragonal symmetry as the major phase. It is reported that processing of anatase TiO2 powder by using conventional hydrothermal route in the highly alkaline media followed by annealing at suitable temperature leads the crystallization of the nanostructured anatase TiO2 with tetragonal symmetry as major phase [26]. The nanostructures of TiO2 formed in this processing includes nanoparticles (NPs), nanotubes (NTs), nanowires (NWs), nanobelts (N Bs), nanorods (NRs) and nanosheets (NSs). The morphology of as-crystallized nanostructured TiO2 depends upon the reaction temperature in the range of 110 - 220 °C during conventional hydrothermal processing [27]. Further, the crystallization of minor phase along with the major anatase TiO2 phase depends upon the processing temperature of conventional hydrothermal route. It is reported that, the processing done at temperature of autoclave in the range of 110 - 170 °C leads the crystallization of sodium titanate (Na2Ti3O7) as minor phase.

Figure 1. XRD patterns for as-prepared SP-TiO2-1, CH-TiO2-2 and
CdS-CH-TiO2-3 films

After hydrothermal processing, washing with 0.1 N HCl and DDW leads the amorphous anatase TiO2 with tetragonal symmetry as major phase along with conversion of Na2Ti3O7 into H2Ti3O7 as minor phase [28-29]. Further, drying of this as-washed mixture at 100 °C and then annealing at a temperature more than 400 °C in air ambient transforms the H2Ti3O7 into TiO2 along with major bulk anatase TiO2. Finally, the annealing process leads the single anatase TiO2 phase with tetragonal symmetry. It is also reported that the processing carried out at autoclave temperature greater than 180 °C leads crystallization of TiO2-(B) with monoclinic symmetry as minor phase [30-31]. These observations are also found to be matching with present case. In present case, the XRD pattern of CH-TiO2-2 film also shows the additional weak peaks marked by '*' symbol. These peaks seen at 2θ = 29.5, 44.6, 58.02 and 67.28 are due to (002), (003), (711) and (621) crystal planes of TiO2-(B) (JCPDS card no. 79-1940) with monoclinic symmetry respectively [31]. Moreover, the XRD pattern for CdS-CH-TiO2-3 film shows the additional peaks at 2θ = 26.6, 28.4, 36.5, 51.5 and 65.7° marked by ‘#’ symbol. These additional peaks at 2θ = 26.6, 28.4, 36.5, 51.5, and 65.7° marked by ‘#’ symbol are found to be matching with (002), (101), (102), (112) and (203) reflections of CdS with hexagonal symmetry (JCPDS card no. 01-0780). It indicates and confirms the formation of CdS coated anatase TiO2 in case of CdS-CH-TiO2-3 film as is expected.

UV-visible spectroscopy

The UV-visible absorbance spectra are presented in figure 2(a) for as-prepared SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films. The absorbance spectra are analysed by using relation: α = (αo/hν)(hν - E g)n, where, α is absorption coefficient, αo is constant, h is Planck’s constant, ν is frequency, E g = energy band gap and n = constant (n = 2 and 0.5 for indirect and direct optical transitions respectively). The Tauc plots: (αhν)2 versus hν for SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films are obtained from corresponding absorbance spectra and presented in figure 2(b).

![Figure 2. (a) UV-visible absorbance spectra and resultant (b) Tauc plots for as-prepared SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films](image-url)

From Tauc plots, the band gap energy (E g) values are obtained by extrapolating the straight-line part of each curve to the zero absorption edge at hν axis as shown in the figure 2(b). The energy band gap (E g) values obtained from Tauc plots for SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films are found to be respectively 3.19 eV, 3.53 eV and 2.52 eV. The E g = 3.19 eV corresponding to the absorption around 300 nm obtained for SP-TiO2-1 film is found to be in good concord with value given in literature for anatase phase of TiO2 in wide range of crystalline size [32]. This is due to bulk nature of TiO2 particles of SP-TiO2-1 film. However, higher value of E g = 3.53 eV in case of CH-TiO2-2 is due to nanostructured nature of TiO2. The absorbance spectrum of CdS-CH-TiO2-3 film given in figure 2(a) shows two transitions: (i) one in ultraviolet region of 300 - 400 nm and (ii) another in visible
region of 400 - 600 nm. The first transition in ultraviolet region of 300 - 400 nm corresponds to the nanocrystalline anatase phase of TiO$_2$. The second transition in visible region of 400 - 700 nm is due to nanocrystalline phase of CdS. Hence, the Tauc plot corresponding to absorbance spectrum of CdS-CH-TiO$_2$-3 film shows two $E_g$ values. The $E_g = 2.52$ eV corresponding to the absorption around 510 nm in CdS-CH-TiO$_2$-3 film is due to the CdS particles coated on nanostructured anatase TiO$_2$ [33]. The higher value of band gap $E_g = 2.52$ eV as compared to the value reported bulk CdS is due nano-crystalline nature of as-coated CdS particles [34]. The second band gap value observed to be $E_g = 3.53$ eV is due to the anatase TiO$_2$ phase.

**Field emission scanning electron microscopy (FESEM)**

Figure 3 shows the FESEM image and corresponding particle size distribution of SP-TiO$_2$-1 powder. The FESEM image indicates the presence of soft agglomerates containing the different number of primary particles. The morphology of primary particles as well as agglomerates is observed to be almost spherical. The histogram gives the size distribution of spherical agglomerates over the number of agglomerates. From histogram, it is seen that maximum number of agglomerates are having the size in range of 25-50 nm indicating thereby the nearly uniform size distribution of the agglomerates. The average value of size of spherical agglomerate is observed to be 36.05 nm. It confirms the nanocrystalline nature of SP-TiO$_2$-1 powder/film. It is consistent with the XRD results, wherein nanocrystalline nature of SP-TiO$_2$-1 film is confirmed.

![FESEM image and corresponding particle size distribution of SP-TiO$_2$-1 film](image)

**High resolution transmission electron microscopy (HRTEM)**

Figure 4 gives HRTEM images for CH-TiO$_2$-2 and CdS-CH-TiO$_2$-3 films. Figure 4 (a - b) depicts the HRTEM images for the CH-TiO$_2$-2 film along with selected area electron diffraction (SAED) pattern. It plainly indicates the nanobelts morphology of as-prepared TiO$_2$ powders of CH-TiO$_2$-2 film. The average width of nanobelts is found to be 200 nm. The d values obtained from the SAED pattern given in figure 4(b) are found to be 3.52, 2.38, 2.87 and 1.93 Å$^2$. The d values: 3.52, 2.38 and 1.93 Å$^2$ are found to be matching with the (101), (004) and (200) reflections corresponding to the anatase phase of TiO$_2$ (JCPDS card no. 21-1272) respectively. Further, $d = 2.87$ Å$^2$ is found to be matching with (002) reflection corresponding monoclinic TiO$_2$-B phase (JCPDS card no. 79-1940). It confirms the formation of anatase TiO$_2$ phase in CH-TiO$_2$-2 film with nanobelts morphology. Figure 4 (c - d) depicts the HRTEM image for the CdS-CH-TiO$_2$-3 film along with SAED pattern. Figure 4(c) show the single nanobelt of TiO$_2$ with number of primary particles deposited on the surface of it. The entire surface of TiO$_2$ nanobelt is observed to be covered with primary particles having hexagonal morphology. The d values obtained from the SAED pattern given in figure 4(d) are found to be 3.52, 2.42, 1.93, 1.51 and 1.37 Å$^2$. The d values: 3.53, 1.93 and 1.51 Å$^2$ are found to be matching with the (101), (200) and (204) peaks corresponding to the anatase phase of TiO$_2$ (JCPDS card no. 21-1272) respectively. Further, d values: 2.42 and 1.37 Å$^2$ are found to be matching with the (102) and (203) reflections corresponding to the CdS with hexagonal symmetry (JCPDS card no. 01-0780). It indicates and confirms the crystallization of CdS particles on anatase TiO$_2$ nanobelts in case of CdS-CH-TiO$_2$-3 film as is expected. The maximum number of CdS particles with hexagonal morphology are found to
be having size in range of 12 - 20 nm. The average value of size of CdS nanoparticles is observed to be 15.86 nm. All above observations related to HRTEM with SAED are found to be consistent with results obtained in XRD and UV-visible spectroscopy studies.

DSSC properties

The J (current density) - V (voltage) characteristic plots recorded for N719 sensitized solar cell assemblies obtained by using the photoanodes of SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 films are presented in the figure 4(e). The nature of curves is similar to J (current density) - V (voltage) curve of standard solar cell. The solar cell properties obtained for different photoanode films are given in Table 1. The values of different solar cell parameters: $I_{SC}$, $J_{SC}$, $V_{OC}$, $I_{MAX}$, $J_{MAX}$, $V_{MAX}$, FF, and $\eta$ are found to be increasing in order of photoanode films: SP-TiO2-1, CH-TiO2-2, and CdS-CH-TiO2-3. The photoconversion efficiency values ($\eta$ %) for SP-TiO2-1, CH-TiO2-2 and CdS-CH-TiO2-3 photoanode films are found to be 0.72, 1.41 and 1.90 respectively. The photoconversion efficiency is found to be increased by 95.83 % in case of CH-TiO2-2 photoanode having nanobelt morphology as compared to the SP-TiO2-1 photoanode having bare TiO2 with spherical morphology. Further, the photoconversion efficiency is found to be increased by 34.75 % in case of CdS-CH-TiO2-3 photoanode having TiO2 nanobelts coated with hexagonal CdS nanoparticles as compared to the CH-TiO2-2 photoanode having bare TiO2 with nanobelt morphology [35-36].

![Figure 4. (a - b) and (c - d) HRTEM images (with SAED patterns) for CH-TiO2-2 and CdS-CH-TiO2-3 film respectively and figure 4 (e) J-V curves for DSSC assemblies fabricated by using SP-TiO2-1, CH-TiO2-2, and CdS-CH-TiO2-3 photoanode films](image)

| Photoanode film | $I_{SC}$ (mA) | $J_{SC}$ (mA/cm²) | $V_{OC}$ (V) | $I_{MAX}$ (mA) | $J_{MAX}$ (mA/cm²) | $V_{MAX}$ (V) | FF | $\eta$ (%) |
|-----------------|--------------|------------------|-------------|--------------|------------------|-------------|----|----------|
| SP-TiO2-1       | 0.31         | 1.96             | 0.607       | 0.24         | 0.11             | 0.44        | 0.609 | 0.72     |
| CH-TiO2-2       | 0.68         | 4.27             | 0.698       | 2.88         | 1.41             | 0.49        | 0.608 | 1.41     |
| CdS-CH-TiO2-3   | 0.76         | 4.75             | 0.682       | 3.89         | 1.90             | 0.50        | 0.586 | 1.90     |

4. Conclusion
The preparation of phase pure anatase TiO$_2$ powders with nanocrystalline spherical and nanobelt morphologies by using basic precipitation method and conventional hydrothermal processing is found to be simple and easy respectively. The minor impurity corresponding to monoclinic TiO$_2$-(B) might be due to high autoclave processing temperature of 190 °C in hydrothermal reaction. Further, method of coating of TiO$_2$ nanobelts with phase pure CdS nanoparticles by using dip coating method is also simple, easy to operate, very low temperature processing and energy-time saving. The novel nanobelt morphology of hydrothermally derived anatase TiO$_2$ useful for enhancing the DSSC efficiency of corresponding of photoanode might be due to presence of nanobelts facilitating the reduction in charge transfer resistance thereby limiting the recombination rate. Further, enhancement of photoconversion efficiency in case of CdS coated TiO$_2$ nanobelt photoanode is due to blocking of back scattered electrons and effective electron-hole charge separation fashioned by co-sensitizer CdS nanoparticles leading to increase in charge transfer current density (J) and reduction in electron recombination rate.

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