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SIMULTANEOUS GROWTH OF RUTILE TiO_2 NANOROD ON FTO PLATE BY ONE-STEP HYDROTHERMAL PROCESS FOR CdS SENSITIZED SOLAR CELL APPLICATIONS

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ABSTRACT

The rutile TiO_2 of nanoparticles and microrods were simultaneously grown on FTO glass slide using a one-step hydrothermal process. CdS semiconducting nanocrystals were deposited on the surface of TiO_2 surface using SILAR method. Moreover, the SEM micrograph studies, TiO_2 nanorods were almost uniformly explored on FTO glass slide pattern. The photovoltaic performances, as-prepared semiconductor sensitized solar cells (SSSCs) were studied and the maximum efficiency of FTO/TiO_2/CdS-8/ZnS-2 sample was 0.78% were confirmed by sun solar simulator, overall the solar cell efficiency was determined with an increasing pattern of CdS deposition is reported in detail.

Contribution/ Originality: In this work, contribute to the development of $Ti0_2$ unidirectional growth nanorods on FTO glass slide and its act as a photoanode was prepared hydrothermal method. The semiconductor (CdS) quantum dots were prepared using SILAR method. These quantum dots acts as dye molecule for the fabrication solarcell. In the present studies gives an idea of semiconductor sensitized solar cells (SSSCs).

1. INTRODUCTION

In the past few years, semiconductor sensitized solar cells (SSSCs) had a great deal of attention as a candidate for third generation photovoltaic devices for the modern electronic era [1]. The focusing of (SSSCs) is similarly like dye-sensitized solar cells (DSSCs), but it was replaced with semiconductor nanocrystals as a sensitizer for light harvesting process [2]. Likely, metal oxide nanostructured were unique properties like tunable bandgap high extinction coefficient and multiple electron-hole pair generation per incident photon, among these terms are increasing the overall performance of solar cell activities [3]. Mostly, metal chalcogenides such as CdS, CdSe, PbS and CdTe, have been investigated as a sensitizing material for SSSCs [2]. Among them CdS has been a most effective sensitizer because of the suitable bandgap, simple fabrication, long lifetimes and excellent stability also reported apparently [3, 4]. In such case, (SSSCs), photoanode is also one of the main components similarly to the

sensitizer. However, the TiO_2, SnO_2 and ZnO nanoparticles have been used as a photoanode materials likely, the power conversion efficiency (PCE) of solar cell depends on size and structure of photoanode matrix [3]. In recent research is focused on the mixing larger grain size of 2D nanostructures like nanorod, nanoflower, dendritic microsphere, microrod, nanowires, etc., Furthermore, TiO_2 nanoparticles to increasing better light harvesting and higher electron transport studies in DSSCs and now in this work, extending this idea to prepare SSSCs applications [5]. Before discussion, Buatong, et al. [6] group has grown the three-dimensional flower-like TiO_2 structure on array of TiO_2 nanorod using double-step by hydrothermal method Buatong, et al. [6]. Cheng, et al. [7] well developed by hydrothermally to construct the structure of photoanode was hierarchical micro-corollas layer upon the nanoforest layer (HTCF) which coronal structures, on the surface of high density and long branched Cheng, et al. [7]. Park and Dhayal [5] has reported simultaneous growth of rutile TiO_2 of 1D/3D-nanorod/nanoflower structure on FTO was constructed in one-step hydrothermal process eventually Park and Dhayal [5]. Sun, et al. [8] also highlighted the dendritic microsphere was deposited on the surface of one-dimensional (1-D) nanowire, TiO_2 nanostructures was also prepared using one-step process on FTO plate [8]. Similarly, Ramalingam, et al. [9] have synthesized CdSe nanorods have been synthesized by the solvothermal method employing cheaper chemicals and easy process, and with better control over the morphology and crystalline quality.

More specifically, Liu, et al. [10] was described the TiO_2 films with trilayer structure (microflowers, nanorod array and compact nanoparticulate) are grown on FTO substrate by one-step hydrothermal approach [10]. In this work, nanoparticles or microrods were simultaneously grown on FTO glass slide as a TiO_2 photoanode by one-step hydrothermal method as per previous literature. Earlier there was no report based on this SILAR method, we are the one first time will be reporting based on the CdS semiconducting nanocrystals were deposited on the surface of TiO_2 using SILAR technique, Moreover, the performances of fabricated solar cells were studied extensively.

2. EXPERIMENTAL SECTION

2.1. Materials

Tetrabutyltitanate [Ti(CH2CH2CH2CH3)4], fluorine doped tin oxide coated glass slide (FTO, Surface resistivity), hydrochloric acid (HCL), sodium sulfide hydrate (Na2S. xH2O) and Sulfur (S) were purchased from Sigma Aldrich. Absolute ethanol (C2H2OH) was obtained from Merck, India. Cadmium nitrate tetrahydrate [Cd(NO3)2. 4H2O] cupric nitrate trihydrate [(Cu(NO3)2. 3H2O] and zinc acetate dihydrate [(Zn(CH3OO)2. 2H2O] and methanol (CH3 OH) were purchased from SRL, India. All reagents were used as received without further purification. In the entire synthesis process deionized water was used as a universal solvent.

2.2. Preparation of Tio2 Thin Film

In stage one, the synthesis of TiO_2 thin film on conducting the surface of FTO glass slide via hydrothermal method. In this process, 1:1 ratio of water (H_2O) and hydrochloric acid (HCL) was taken in the capacity of 40 ml of both the mixture and kept it at uninterrupted stirring for 20 min at room temperature. After that 1.5 ml of tetrabutyltitanate was added into above solution for 15 min stirring. In this reaction the milky white color was tuned into transparent solution respectively. In stage two, FTO glass slide were taken and washed with 1:1 ratio of methanol and acetone for 20 min using probe type sonicator. Finally, as-prepared precursor (stage one) has been transferred into Teflon coated autoclave, the FTO glass slide was kept under the bottom of autoclave. The entire reaction undergoes by hydrothermal treatment at 160 ˚C for 14 hrs. The autoclave was cooled naturally to reach room temperature and rinsed with deionized water. Finally, the sample were calcined at 500 ˚C for 15 min and labelled it as FTO/TiO_2.

2.3. Deposition of CdS Sensitizer

Cadmium sulfide (CdS) were deposited on the surface of TiO_2 by successive ionic layer adsorption and reaction (SILAR) method. In First stage, the FTO/TiO_2 thin film was dipped in cationic ethanol solution of 0.4 M of cadmium nitrate tetrahydrate for 4 min and rinsed with ethanol. In second stage, the rinsed film was dipped into anionic methanol solution of 0.4 M sodium sulfide hydrate and rinsed with methanol solution. The objective of the above two processes was defined as one cycle and repeated several times. The prepared sample was called as TiO_2/CdS-4 and TiO_2/CdS-8 for 8 SILAR method [11].

2.4. Deposition of Passive Layer

ZnS passive layer was deposited by using SILAR method. Very firstly, the FTO/TiO_2/CdS thin film was dipped in 0.1 M of cationic aqueous zinc acetate dehydrate solution for 4 min and 0.1 M of anionic aqueous sodium sulfide hydrate solution for 4 min, then rinsed with ethanol and using dryer to dry the sample and it follow SILAR reaction. The photoelectrodes were so-called as FTO/TiO_2/CdS-4/ZnS-2 and FTO/TiO_2/CdS-8/ZnS-2. Finally, all the samples were annealed at 80 °C (1 hrs) to remove the solvent as well to make crystallinity of the sample [12].

2.5. Counter Electrode Preparation

In the typical synthesis, 2 M of cupric nitrate trihydrate is taken in 10 ml of de-ionized water and 2 M sodium sulfide hydrate with 10 ml of water. Mix both precursors together and stirred it well for 5 min and keep it stable for 2h at room temperature. Finally, it was centrifuged with ethanol, collected as a nanopowder, and dry it at 100 °C for 3h [13]. 0.0025 g of prepared CuS power was taken in 10 ml ethanol and sonicated it 30 min. It was filled in spray container and sprayed it on FTO glass slide with substrate temperature was 70 °C for 20 min. Finally, the resultant sample was annealed at 80°C for 30 min.

2.6. Device Assembly

In this work, solar cell fabrication procedure was followed by the literature [14] and detailed process was given below. The photosensitizing anode and counter electrode was sandwich together using a scotch tape spacer with binder clip and few drop of polysulfide electrolyte was injected in between them. The polysulfide electrolyte solution makes 2 M of sodium sulfide hydrate and 2 M of sulfur in water-methanol (7:3, v/v) solution. A black mask on the photoanode with a window of 0.25 cm^2 was used to select photo active area of the cell [15].

2.7. Characterization Studies

The crystal phases of the films were studied by the help of X-ray Diffraction (XRD, PANalyticalX'pert Pro) with Cu k_α and 2θ range from 10° to 80°. The morphologies were examined by scanning electron microscopy (SEM, VEGA 3 TESCAN) with energy dispersive X-ray (EDX) spectrum (Bruker). The optical properties of the as-prepared samples were performed by UV-Vis-NIR Spectrophotometer (Ocean optics HR 2000). The Photovoltaic studies (J-V curves) of CdS sensitized solar cells were measured using Agilent B2901A precision source/measure meter under one sun solar LED simulator (Royal Enterprises, Chennai, Model:100L). The intensity of the simulator was setup to 100 mW/cm^2 condition using standard silicon reference cell.

3. RESULT AND DISCUSSION

3.1. XRD Analysis

The XRD patterns of FTO/TiO_2 and FTO/TiO2/CdS-8 thin film samples system were recorded at 2θ range between 10° to 80° as shown in Figure 1. The diffraction peaks are located at 27.8°, 36.3°, 41.4°, 54.5°, 56.7°, 62.8°, 65.6°, 68.9° and 69.8° with respective plane of indices as (110), (101), (111), (211), (220), (002), (221), (301) and

(112). The observed peak of synthesized samples confirms the tetragonal phase of rutile TiO_2 and its followed the JCPDS file (Card No: 65-0192) [16]. The diffraction peaks located at 25.1° and 43.9° with indices as (100) and (110) corresponding to CdS sensitizer phase (JCPDS Card No: 65–3414) as shown in the Figure 1b [17]. Further this reveals that the diffraction peaks of CdS phase were absent, because most of the peaks are very near to SnO_2 peaks. Further there is no other unwanted peak observed in presented sample.

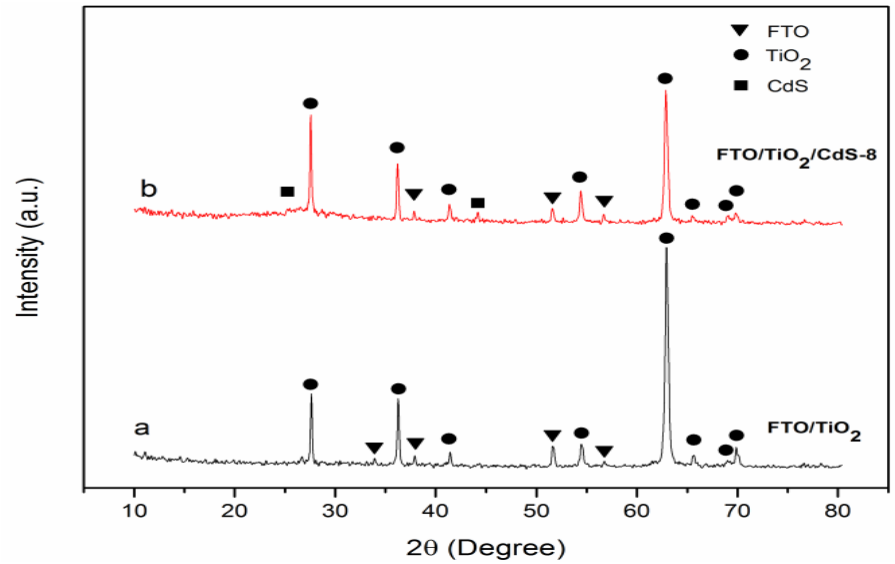

Figure-1. XRD patterns of (a) FTO/TiO2 thin film and (b) FTO/TiO2/ CdS-8 thin film.
Source: OriginPr8.

3.2. Raman Spectroscopy Studies

The Raman Spectra of FTO/TiO_2 and FTO/TiO_2/CdS film were shown in the Figure 2. In the spectrum of FTO/TiO_2 thin film, the peaks at 235, 441 and 607 cm^{-1} are typical rutile TiO_2 bands and these can be corresponding to the two-photo scattering, E_g and A_{1g} modes in Figure 2a [18, 19]. The strong peak found at 298 cm^{-1} which can be recognized to the first-order LO phonon vibrational modes of CdS in FTO/TiO_2/CdS-8 sample [20, 21]. So, the results from Raman spectra confirm that TiO_2 have in rutile phase with CdS crystal and are in good agreement with the XRD result.

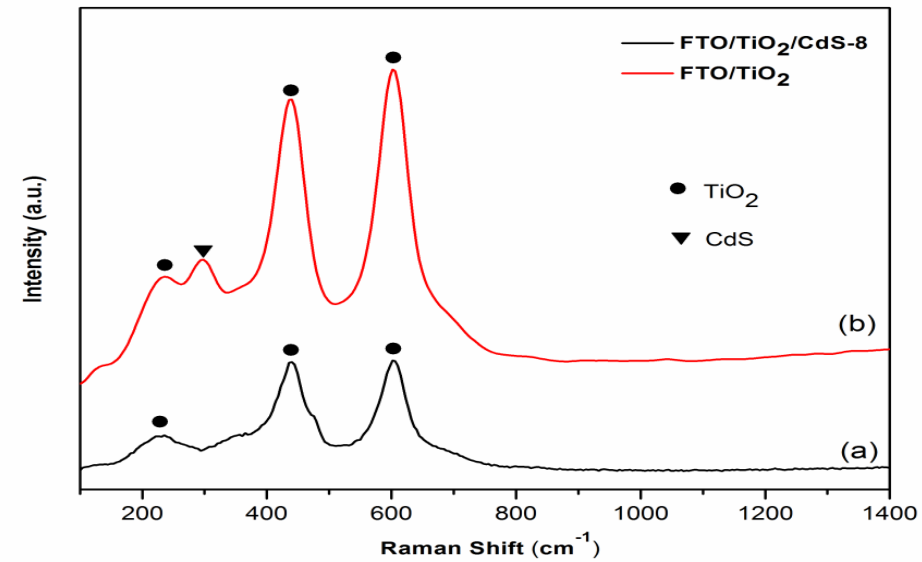

Figure-2. Raman spectra of (red curve) FTO/TiO2 thin film and (black curve) FTO/TiO2/ CdS-8 thin film.
Source: OriginPr8.

3.3. Morphological Studies

Figure 3a-c, shows the SEM micrographsof FTO/TiO_2 thin film with different magnification. In these SEM micrographs, microrods as well as nanoparticles of rutile TiO_2 are grown on FTO glass slide in one-spot hydrothermal process. In Figure 3c, TiO_2 nanoparticles are uniformly deposited on the conducting surface of FTO glass slide with diameter is approximately 200 nm. In Figure 3b, gives the novel exploration of TiO_2 microrods taken place on FTO glass slide is almost uniformed manner. The diameter and length of the microrods is 50 nm and 30 nm. The thin layer of CdS nanocrystals were deposited on the surface of microrods and nanoparticles as shown in Figure 4a-c. The Figure 4d&e, gives the EDX spectra of FTO/TiO_2/CdS-8 to study the compositional details of the sample. In EDX studies, CdS crystals were deposited more on microrods surface compare to nanoparticles, because of TiO_2 microrods have high surface area. The EDX results give agreement with both Raman spectra and XRD results.

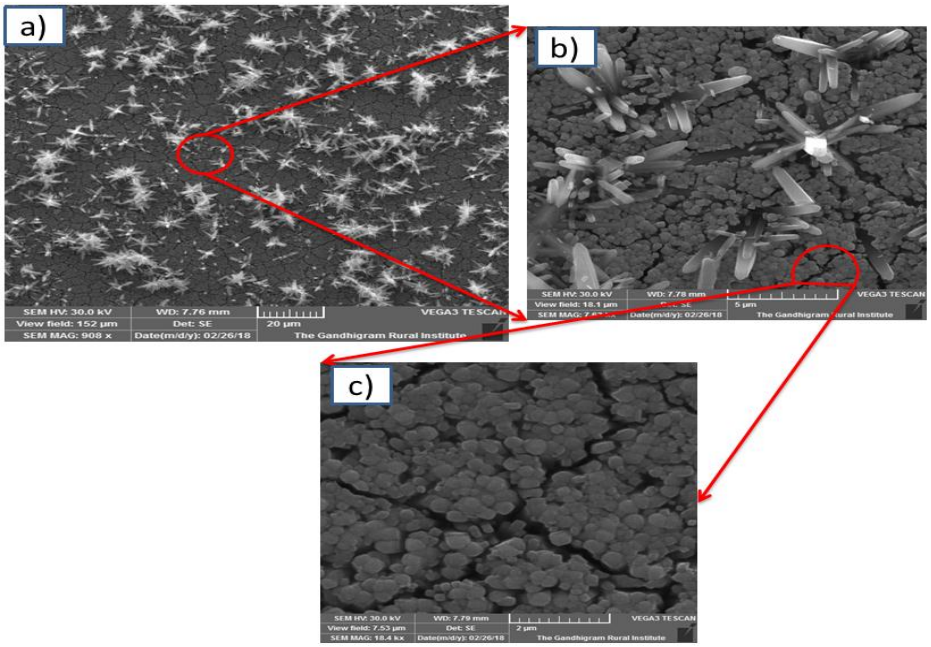

Figure-3. SEM images of FTO/TiO2 thin film.

Source: Jpeg image.

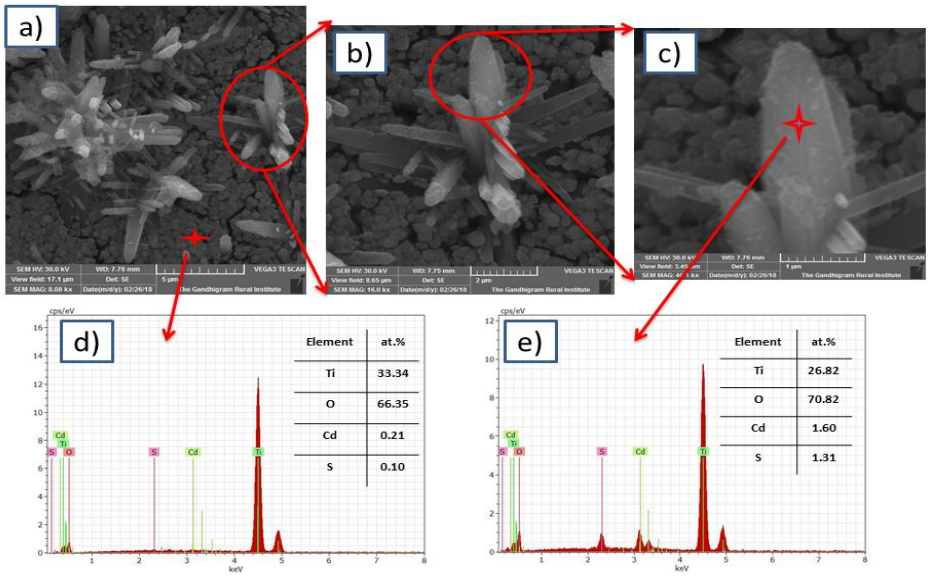

Figure-4. SEM and EDX images of FTO/TiO2/CdS-8 thin film.

Source: Jpeg image.

3.4. Optical Studies

The absorption spectra of FTO/TiO_2 and FTO/TiO_2/CdS-8 thin film is shown in Figure 5. From these spectra, FTO/TiO_2/CdS-8 thin film has higher absorption intensity in the visible region compare to FTO/TiO_2. The absorption spectra of FTO/TiO_2 thin film shows broad absorption band and absorption edge is 408 nm and corresponding bandgap of ~3.4 eV. After deposition of CdS sensitizer on FTO/TiO_2, the absorption edge was shifted (red shift) from 408 to 521 nm in visible range and its corresponding bandgap of 2.7 eV (521). The band gap of bulk CdS is ~2.42 eV from the literature [9]. The bandgap of FTO/TiO_2/CdS-8 is higher than bulk, so it's indicated that CdS nanocrystals are obey the quantum confinement effect [22].

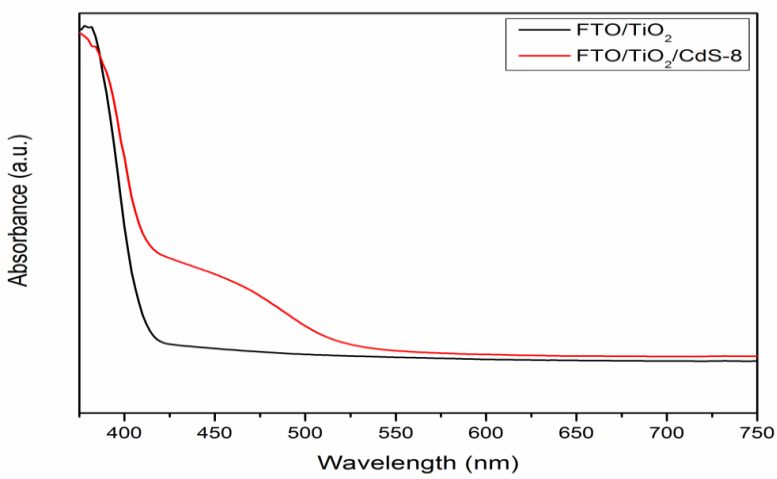

Figure-5. UV-Vis spectrum of a) FTO/TiO2 thin film, and b) FTO/TiO2/CdS-8 thin film.
Source: OriginPr8.

A schematic diagram of CdS sensitized solar cell structure with expected pathway of charge transfer as shown in the Figure 6. The solar cell consists of semiconducting TiO_2 on FTO, CdS sensitizer, ZnS passive layer, polysulfide electrolyte (S_n^{2-}/S^{2-}), and CuS Counter electrode (CEs) on FTO. The advantage of ZnS passive layer is to improve the cell performance, because of reduced charge recombination. The conduction band of ZnS is higher when compare to CdS, so the generated charge carrier have quantum electrical confinement effect [23]. During cell working process, CdS sensitizer with ZnS passive layer absorbs the photons to create electron-hole pairs and these pairs are separated into electrons and holes intermediate between semiconductor (TiO_2) and sensitizers. The electrons are travel into the semiconducting TiO_2 and then to FTO substrate and finally enter the external circuit. These electrons passed through the counterpart and then, enter the electrolyte and the process is repeated.

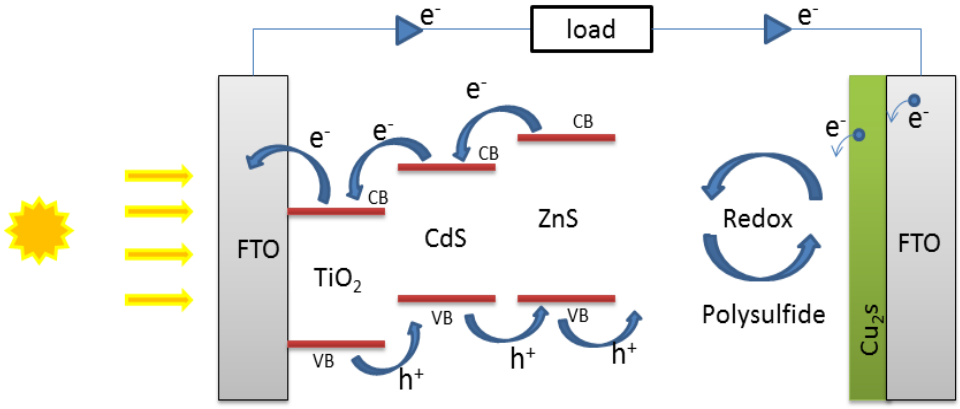

Figure-6. Performance of CdS sensitized solar cell based on CuS Counter electrode.
Source: Jpeg image.

3.5. J-V Characterization Studies

Figure 7 shows the photocurrent density - photovoltage (J-V) performance of fabricated CdS sensitized solar cell module under solar simulator with one sun radiation. The short circuit current density (J_{sc}), open circuit voltage (V_{oc}), Fill Factor (FF) and efficiency are the solar cell parameters for fabricated SSSCs which are summarized in Table 1. The FTO/TiO2/CdS-8 solar cell has fill factor(FF) and efficiency about 32.9% and 0.49%. From the Table 1, as prepared CdS sensitized solar cell efficiency depends on the increasing layer of CdS deposition. In ZnS passivation layer on FTO/TiO_2/CdS-8, the efficiency increased from 0.49 to 0.78%, because of greatly eliminated interfacial charge recombination to improve the efficiency of the solar cell [4]. In comparison, it is concluded that from the entire four samples, the FTO/TiO_2/CdS-8/ZnS-2 sample have greater efficiency in the active area of 0.25 cm^2.

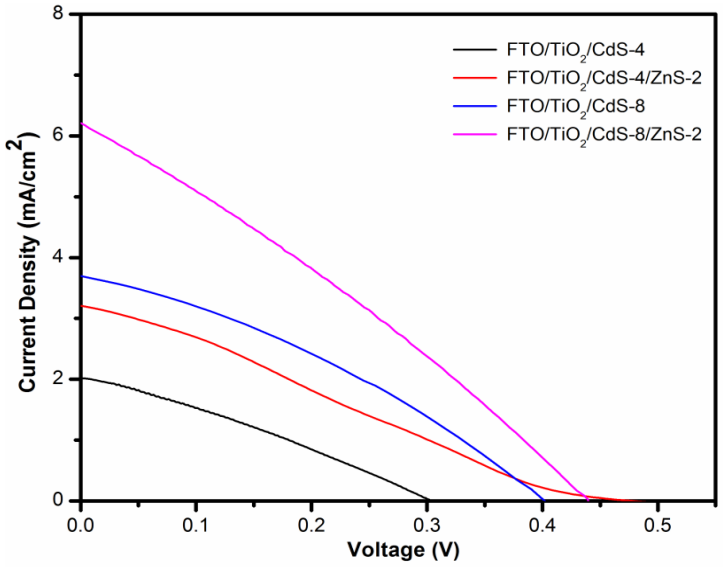

Figure-7. J-V curve of as-prepared CdS sensitized solar cell.
Source: OriginPr8.

Table-1. Solar cell parameter of as prepared samples.

Sample code	V_{oc}(V)	J_{sc}(mA/ cm^2)	FF%	η%
FTO/TiO_2/CdS-4	0.304	2.01	30.0	0.18
FTO/TiO_2/CdS-4/ZnS-2	0.478	3.20	23.7	0.36
FTO/TiO_2/CdS-8/	0.402	3.70	32.9	0.49
FTO/TiO_2/CdS-8/ZnS-2	0.440	6.21	28.7	0.78

Source: MS office word doc.

4. CONCLUSION

In this summary, the successful growth of nanoparticles and microrods simultaneously on FTO glass slide as a TiO_2 photoanode by one-step hydrothermal method is achieved. The CdS was deposited on TiO_2/FTO glass slide to sensitize the film in visible region. The SEM results confirmed that, the TiO_2 microrods were explored on FTO glass slide uniformly. The efficiency of fabricated cell increased due to increase in the layer of deposition of CdS on TiO_2/FTO. The efficiency of FTO/TiO_2/CdS-8 sample was increased from 0.49% to 0.78% due to the deposition of ZnS passive layer (FTO/TiO_2/CdS-8/ZnS-2).

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Contributors/Acknowledgement: All authors contributed equally to the conception and design of the study.

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