Nano-Structures & Nano-Objects 19 (2019) 100311

Contents lists available at ScienceDirect

Nano-Structures & Nano-Objects

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

Novel stainless steel based, eco-friendly dye-sensitized solar cells using electrospun porous ZnO nanofibers

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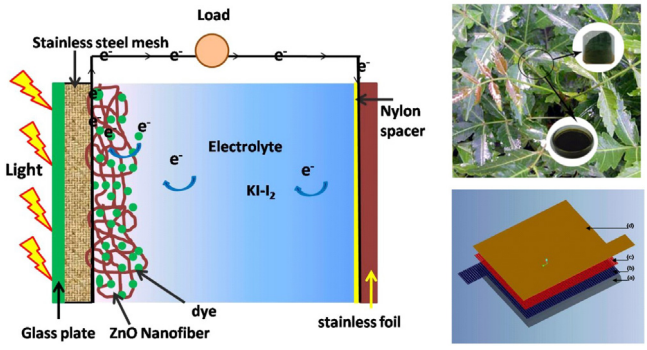
GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 31 October 2018
Received in revised form 31 March 2019
Accepted 15 April 2019

Keywords:
Dye-sensitized solar cells
Electrospinning
ZnO nanofibers
Chlorophyll
Natural dyes

ABSTRACT

Herein, we report a new type of eco-friendly, cost-effective stainless steel mesh-based flexible quasi-solid dye-sensitized solar cell (DSSC) using electrospun ZnO nanofibers as the photoelectrode. The electrospun ZnO nanofibers showed enhanced surface to volume ratio due to the high porosity of the fibers composed of ZnO grains having 12–20 nm size with high interconnectivity. For DSSCs fabrication, commonly available natural dye chlorophyll was extracted from the Neem plant and used as the dye sensitizer. An overall solar cell conversion efficiency of 0.13 % was observed for the assembled DSSC with a short-circuit photocurrent, open-circuit voltage and fill factor of 28 μA, 0.321 mV and 32.77 % respectively. The present approach to develop cost-effective and eco-friendly DSSCs would open up enormous possibilities in effective harvesting of solar energy for commercial and rural area applications

1. Introduction

Development of efficient and low-cost technologies based on dye-sensitized solar cells (DSSCs) promise enormous scope and potential in harvesting solar energy [1–3]. DSSCs have a great potential to be an alternative device for the expensive conventional photovoltaics due to their low production costs and potentially high conversion efficiency, reaching 11% and a module efficiency of 7% in few cases [4]. For realizing high efficiency in DSSCs, effective adhesion of semi-conductor metal oxide layer to the transparent conducting oxide coated substrate, a very high surface area for the porous semiconductor metal oxide thin film for efficient absorption of the sensitizer, and proper necking (connectivity) between the particles are essential. The development of conventional low-temperature fabrication methods

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https://doi.org/10.1016/j.nanoso.2019.100311
2352-507X/

should overcome two major problems of incomplete necking of the particles and the presence of residual organics in the film which cause lower electron diffusion coefficients and electron lifetimes. Although several approaches are reported in literature, large-scale synthesis of such nanostructures is difficult from the perspective of commercialization [5,6]. The standard approach for manufacturing DSSCs is to fabricate them onto conducting glass substrates [7]. Due to the difficulty in large scale manufacturing of such DSSCs on glass substrates, researchers started thinking of alternative flexible substrates instead of glass. Recently, some efforts have been made to develop methods compatible with flexible polymeric substrates [8,9]. In this scenario, electrospinning is a versatile technique for the mass production of metal oxide nanofibers having large surface area which can be used for the fabrication of DSSCs [10].

Amiri et al. reported a cooperative quantum dot and plasmonic effect on improving the performance of dye synthesized solar cells by incorporating CdS QDs, gold nanoparticles (GNPs), gold nanorods (GNRs) and Au-decorated Ag dendrites into the active layer [8,11]. Kamazani et al. introduced CdIS-QDs as barrier layer in dye sensitized solar cells (DSSCs). Researchers developed inexpensive solar cells with Se, Te and $CuInS_2$ nanostructures using microwave and hydrothermal synthesis [12–16]. DSSCs with high conversion efficiency were reported with TiO_2, ZnO and Ag@organic nanostructures synthesized using primary amine and tripodal tetramine ligand [14,17–20]. Teymourinia et al. developed a facile synthesis of graphene quantum dots from corn powder for DSSC [21–24]. Sabet et al. investigated different chemical methods for deposition of CdS on TiO_2 surface for the application of DSSC [25].

Many efforts have focused on sensitizer dyes also, since dye plays a key role in harvesting sunlight and transferring solar energy into electric energy. So far, several synthetic organic dyes and organometallic complexes have been employed to sensitize nanocrystalline semiconductors [26–28]. One of the most popular efficient sensitizer is transition metal coordination compound, ruthenium polypyridyl complex due to their intense charge-transfer (CT) absorption in the whole visible range, long excited lifetime and highly efficient metal-to-ligand charge transfer (MLCT) [29]. But, due to the complexity in synthesis of such synthetic organic dyes, researchers are in search of useful dyes which are abundant in natural products for the fabrication of DSSCs [30,31]. Because of the simple extraction techniques and cheap cost, natural dyes are promising candidates as alternative sensitizers for dye-sensitized solar cells [32,33].

In conventional DSSCs, expensive platinum thin films formed on transparent conductive glass substrates by vacuum vapor deposition or sputtering are used as the counter electrodes of DSSC [34,35]. Therefore, in order to reduce production cost of the cells, in this investigation, we have used flexible stainless steel mesh instead of the transparent conducting oxide glass substrate and stainless steel foil instead of Pt coated glass substrate. Stainless steel electrodes increases electrical conductivity by lowering the series resistance of the cells and thus increase the overall efficiency, especially when the cell area increases [36]. Stainless steel electrodes also lower the risk of electrolyte leakage and material contamination by possible fractures in the glass. In this investigation, we report a novel eco-friendly and cost-effective fabrication technique for the production of DSSCs based on stainless steel. Apart from that, for this study, a widely available natural dye, Chlorophyll, extracted from Neem (*Azadirachta indica*) leaves was used as the dye sensitizer for the fabrication of the DSSCs.

2. Experimental section

2.1. Electrospinning of ZnO nanofibers

ZnO nanofibers based porous photoelectrode was fabricated by using electrospinning technique [37,38]. The feed solution for the ZnO nanofiber production was prepared by blending Zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$, MERCK) and PVA (HIMEDIA) in 29 mL of ultrapure water (Millipore Academic, Resistivity, 18.2 MΩ cm) and the solution was stirred for 3 h at room temperature. 1 mL of ethanol (MERCK, 99.9%) of was then added to the solution and stirred for 30 min for homogeneity. A clear solution thus obtained was then subjected to electrospinning at a voltage, 26 kV with a feed rate of 0.2 mL/h. The distance between the collector and needle was optimized to be 12 cm. The stainless steel mesh (SS 304 grade) were cut into pieces and washed with water and mild detergent. Cleaning was completed by sonication in an ultrasonic bath for 30 min in Millipore water. Finally, the stainless steel meshes were dried under dry nitrogen purging and thermal treatment was carried out in a furnace for 600 °C in 30 min. A 21G needle was used for electrospinning. The electrospun fibers were collected on the stainless steel mesh. In order to create an interconnected mesoporous semiconducting network, and also remove supporting polymer, atmospheric water and other adsorbents from the ZnO nanofibers, the film was further calcined at 600 °C for 2 h.

2.2. Extraction of chlorophyll from neem leaves

Clean and fresh Neem (*Azadirachta indica*) leaves were used for the extraction of chlorophyll, and were used as the dye sensitizer for the DSSC fabrication. 250 mg of leaf tissue was placed in a vial containing 7 mL of dimethyl sulfoxide (DMSO) and extracted chlorophyll into the DMSO without grinding by incubating overnight at 65 °C. The extracted chlorophyll was transferred to a graduated tube and made up to a total volume of 10 ml with DMSO. The solution was stored under refrigerated conditions until use. The same method was also adopted for chlorophyll extraction using acetone. But, in this case, the leaf tissue was macerated well using a pestle and mortar before extraction using acetone. Fig. 1a shows the photograph of the extracted chlorophyll from Neem leaves.

2.3. Fabrication of stainless steel based DSSCs

Fig. 1B shows the overall assembly for the fabrication of stainless steel based cost-effective dye sensitized solar cells which is used for this study. The nanoporous, electrospun ZnO nanofibers fabricated on stainless steel mesh (25 × 25 cm) was used as the photoanode. The photoanode was assembled with a transparent glass substrate (25 × 25 cm) for sunlight exposure onto the photoanode and also to prevent leakage of electrolyte. Chlorophyll, the dye sensitizer was coated over the electrospun ZnO nanofiber by spray coating technique instead of adsorption technique, where, internal impedance of the cell increase with adsorption time [39]. The DSSCs were assembled by filling KI-I_2 electrolyte in ethylene glycol and acetonitrile mixture with 4:1 v/v ratio, in between the electrospun, porous ZnO photoanode and stainless foil counter electrode, which are assembled together. A Nylon spacer was used in between the photoanode and the counter electrode in order to prevent short circuit between the cathode and anode. The edges of the DSSC were sealed using Teflon film in order to prevent leakage of the electrolyte from the cells. Solar simulator Current–voltage measurements were taken using Keithley 2420 source measuring unit (SMU) using 2 probe method and then its corresponding *JV* curve had been derived and the measurements were repeated thrice to ensure its repeatability. The measurements were tested and repeated for morning and evening sunlight to test the efficacy of fabrication.

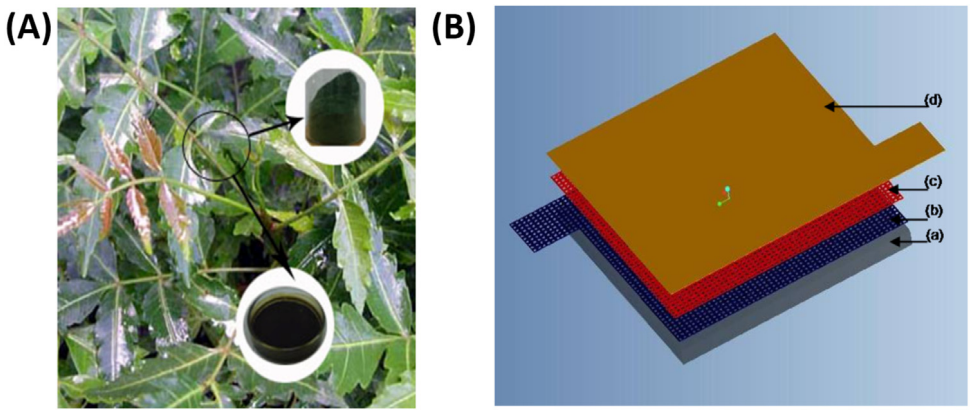

Fig. 1. (a) Photograph of the Chlorophyll extracted from Neem plant, (b) Pro-E Modeling of DSSC (i) Glass substrate (ii) ZnO coated SS mesh (iii) Nylon spacer (iv) SS foil.

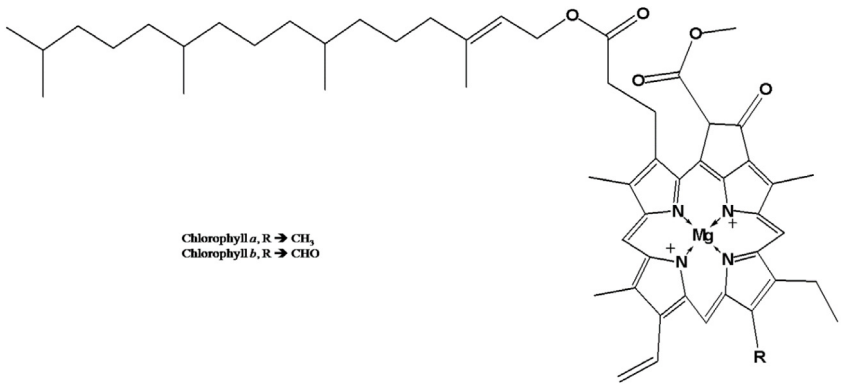

Fig. 2. Molecular structure of Chlorophyll.

2.4. Characterization techniques

The spectral analysis of the chlorophyll extracted from Neem leaves was performed using UV–Vis spectrophotometer (PG Instruments, UK). XRD patterns were acquired using XD-2 Powder X-ray diffractometer (JSO-DEBYEFLEX 2002, Japan) using Cu Kα radiation of wavelength 1.54 nm and with a scanning rate of 0.02 °/s in the 2θ range of 10–70°. Atomic Force Microscopy was performed in semi-contact mode with a NTEGRA Multimode Scanning Probe Microscope (NT-MDT, Russia) for the electrospun ZnO nanofibers before and after calcinations. Transmission Electron Microscopy coupled with selected area diffraction analysis (SAED) was done with a JEOL 2010 Electron Microscope for the transferred ZnO nanofibers onto carbon coated copper grids. The photovoltaic tests of assembled DSSCs were carried out by measuring the *I–V* characteristics curves under irradiation with white light of 1000 LUX in ambient atmosphere.

3. Results and discussion

Chlorophyll is an effective photoreceptor since they contain a network of alternating single and double bonds, and the electrons can delocalize stabilizing the structure as shown in Fig. 2 Such delocalized polyenes have very strong absorption bands in the visible regions of the spectrum, allowing the DSSC to absorb the energy from sunlight. In order to fabricate eco-friendly solar cells, for the current investigation we have chosen chlorophyll as the dye sensitizer.

The purpose of choosing this dye as the photosensitizer is to absorb maximum light energy as possible and inject the photo-excited electrons into the conduction band of the semiconductor material. Fig. 3a shows the UV–VIS absorption spectra of the extracted chlorophyll using DMSO and acetone respectively. From the spectral analysis it was inferred that the chlorophyll extract contained both chlorophyll *a* and *b* as a mixture and the percentage of each type varies depending on the extraction solvent. But, for the Acetone extract, the presence of other carotenoids also was evident (Fig. 3a) and therefore DMSO extract of Chlorophyll was chosen for the DSSC fabrication. The different side groups in both chlorophylls tune the absorption characteristics to slightly different wavelengths, so that light that is not significantly absorbed by chlorophyll *a*, at, say, 460 nm, will instead be captured by chlorophyll *b*, which absorbs strongly at that wavelength [40, 41]. Thus these two kinds of chlorophyll complement each other in absorbing sunlight. Therefore, for this study the mixture of chlorophyll *a* and chlorophyll *b* was chosen, rather than separating both types. Dyes have specific functional groups to get absorbed onto the oxide substrate for evident charge transport process during solar cell fabrication, invariantly esters, hydroxyls, carbonyl and most importantly porphyrins groups were observed for the extracted chlorophyll (Figure. S1).

In order to infer about the porosity of individual nanofibers TEM imaging was performed as shown in Figs. 5 and 6. The morphology of the nanofibers was clearly visible from the TEM image (Fig. 5), with high connectivity and length as required for DSSC fabrication. The nanofiber consist of both polymer supported Zn(OH) composites as its taken before heat treatment.

The electronic spectrum of chlorophyll is characterized by two regions: the *Q* band, which is relatively weak and occurs in the visible region, and the intense *B* band that appears in the near UV region and is often accompanied by an *N* band of lower intensity as shown in Fig. 3. Further Fig. 3b depicts the time

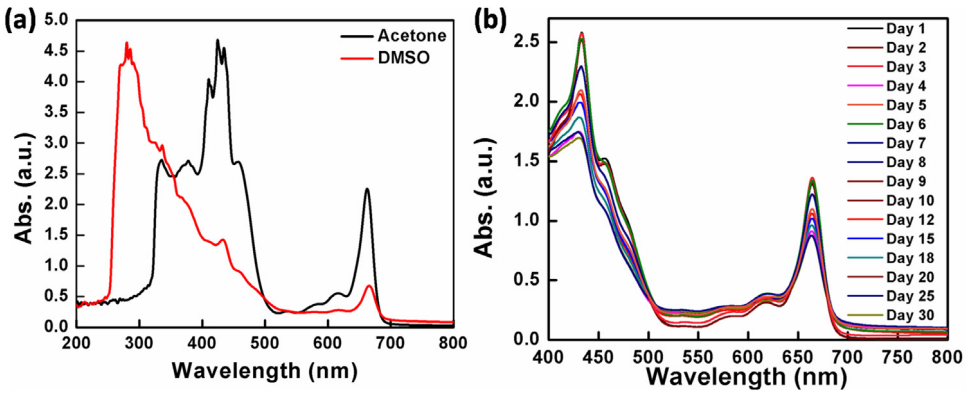

Fig. 3. (a) The absorption spectra of the chlorophyll extracted by acetone and DMSO and (b) spectra of chlorophyll degradation as a function of time up to one month.

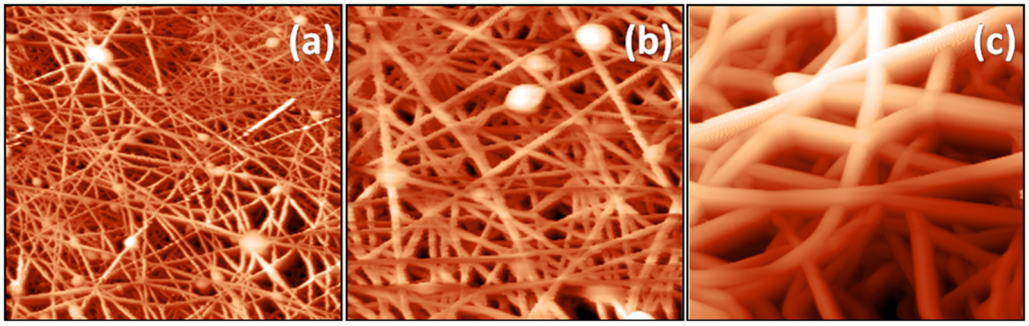

Fig. 4. (a–c) AFM images of the ZnO nanofibers produced by electrospinning.

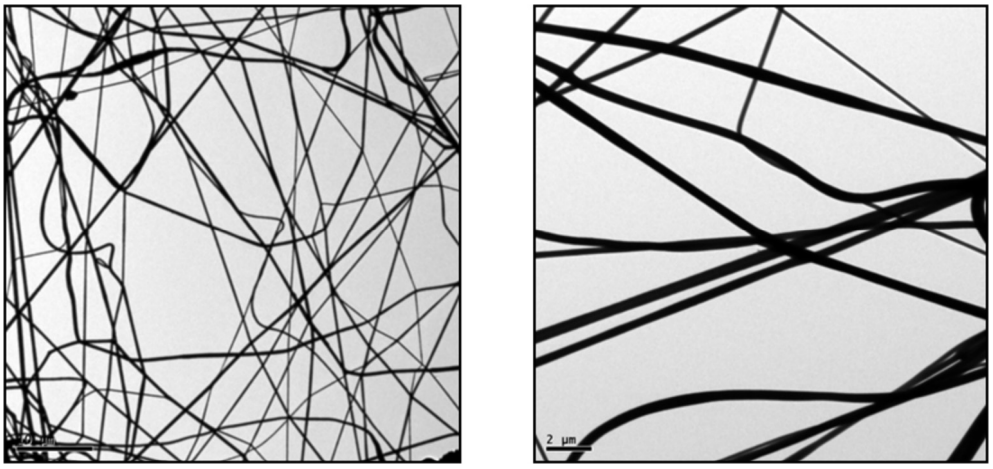

Fig. 5. Representative HRTEM images of the ZnO nanofibers before heat treatment.

dependent absorption characteristics of Chlorophyll indicating the degradation of Chlorophyll as a function of time up to 30 days.

The photoanode was prepared using the versatile technique, electrospinning. Polyvinyl Alcohol (PVA) was used as the supporting polymer along with ZnO precursor solution for the formation of uniform nanofibers. The organic content of PVA was removed after calcinations of the nanofibers at 600 °C. Randomly oriented ZnO nanofibers were found to be continuous having diameters ranging 200–300 nm. Fig. 4 shows the AFM images of the ZnO nanofibers showing the fiber morphology. The nanofibers were found to be porous and grainy due to evaporation of the polymer after heat treatment. Structural morphology and crystallinity of the ZnO nanofibers were studied using XRD analysis as shown in Figure. S2 (See supporting information). Compared to the as-such spun fibers, annealed ZnO nanofibers shows an improved crystallinity with prominent hexagonal wurtzite structure with space ground of $P6_{3mc}$ (186) (a – 3.249 Å and c – 5.206 Å) (JCPDS PDF no 36–1451).

After annealing the nanofibers around 350 °C, TEM studies had been carried out as evident from Fig. 6. HRTEM images of the ZnO nanofibers revealed the presence of highly porous fiber structure [42,43] having ZnO grains with 12–20 nm size with high interconnectivity (vide Fig. 6).

Highly crystalline nature of the ZnO had been observed for the annealed samples with continuous fibers made of smaller nanocrystalline grains. Because of the overlapping of the peaks in the Energy dispersive X-ray (EDS) data (due to very close values

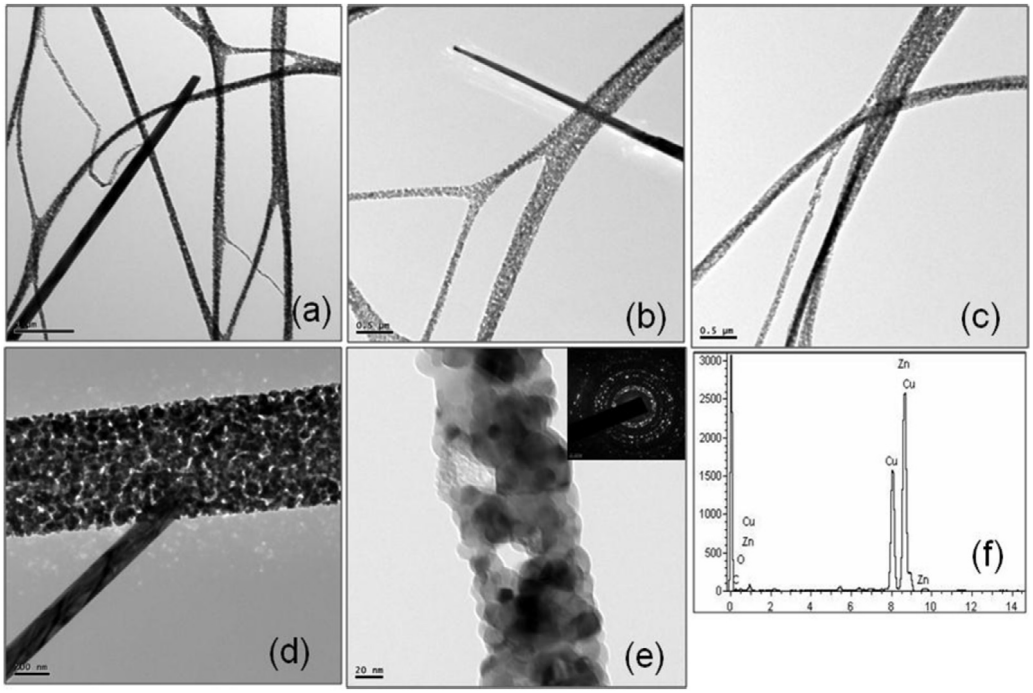

Fig. 6. (a–e) Representative HRTEM images of the heat treated electrospun ZnO nanofibers, (f) EDS spectrum of the ZnO nanofibers.

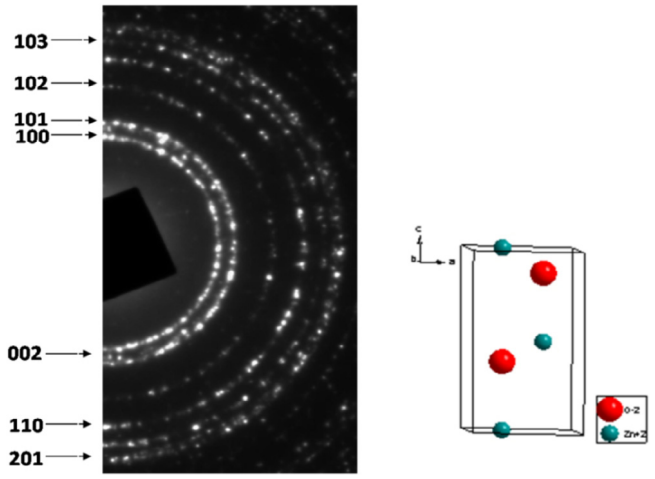

Fig. 7. (a) SAED pattern take from ZnO nanofibres indexed based on hexagonal crystal structure and (b) unit cell of ZnO having Wurtzite crystal structure.

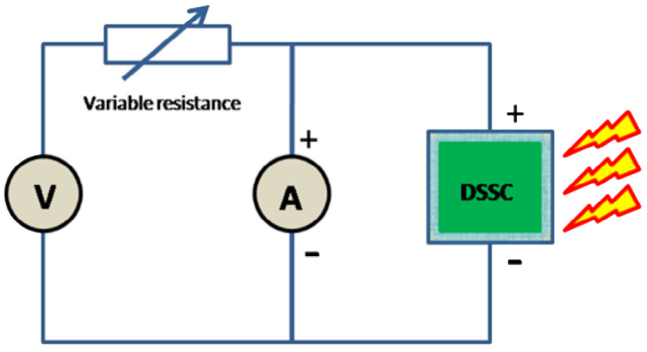

Fig. 8. Schematic circuit diagram of the experimental setup used for measuring the current–voltage characteristics of DSSC with variable resistance potentiometer (1 MΩ), Voltmeter (V) and Ammeter (A).

of X-ray energies for copper and zinc) it is difficult to confirm the presence of ZnO. Selected Area Electron Diffraction (SAED) pattern taken from the nanofibers (shown in Fig. 7) was indexed according to a hexagonal primitive (wurtzite) crystal structure having cell dimensions $a = 3.253$ Å and 5.213 Å (space group $P6_3mc$, JCPDS card no- 89–1397). By comparing the TEM images of the post annealed fibers with AFM images of the continuous and non porous fibers before annealing, it is evident that the polymer parts in between the ZnO nanoparticles in the fiber have evaporated resulting in the formation of a fiber with a highly porous skeleton of ZnO nanoparticles. Moreover, the very high melting point (about 1500–2000 ° C for 15–30 nm particle size) of ZnO nanoparticles and very low boiling point of PVA indicate two facts. First, there is no possibility of reminiscent PVA in the nanofibers after annealing up to 600 °C for 2 h. Secondly there cannot be any primary chemical bonding between two nanoparticles in contact with each other. *i.e*, there is no tendency for the particles to fuse with each other. However, the contact between the nanoparticles could be such that there is electrical continuity from one end of the fiber to the other for the electrons that are produced during the incident light. Though there can be "active centers" in the form of anion and cation vacancies inside the crystal, it is the surface defects that play the major role in the movement of electrons.

The photovoltaic tests of the assembled DSSCs were carried out under irradiation with white light of 1000 LUX (0.00014 W/cm^2) in ambient atmosphere using an experimental setup described in terms of the electronic circuit as depicted in Fig. 8. The photocurrent *vs* voltage (I–V) characteristics curve of the assembled DSSCs based on the stainless steel mesh electrode is shown in Fig. 9a. Assuming our DSSC exhibits a diode like behavior [44],

$$I(V) = I_{ph} - I_S \left[\exp\left(\frac{V}{V_T}\right) - 1 \right] \quad (1)$$

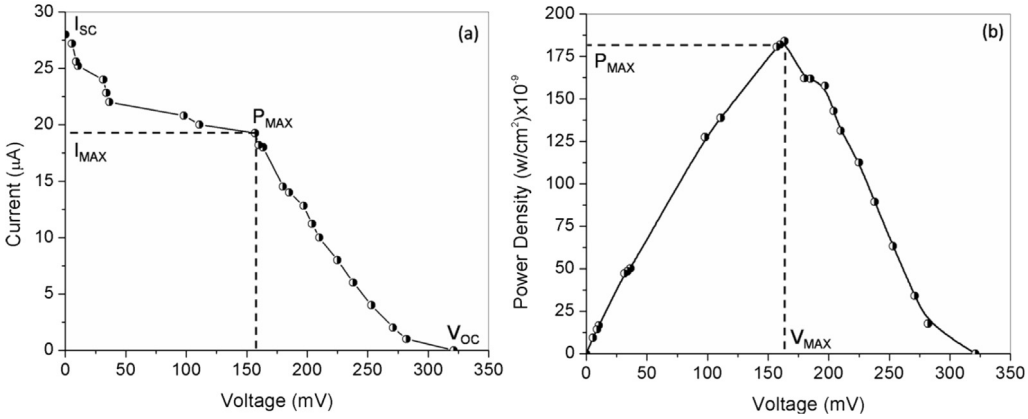

Fig. 9. (a) Photocurrent–Voltage characteristics and (b) current vs. power curve for the assembled stainless steel based DSSC.

where, where I_s is the saturation current of the diode and I_{ph} is the photocurrent, V is the applied voltage and V_T is the thermal voltage. This expression only includes the ideal diode current of the diode, thereby ignoring recombination in the depletion region. From this expression, it is clear that the relationship between output voltage and output current is nonlinear, $I = f(V)$, and Ohms Law will at best be obeyed in a piece-wise fashion. As indicated in Fig. 9a, an important point on the I–V curve is the point at which maximum power is supplied which is also referred as the 'knee of the curve'. Point (i) $I = I_{SC}$ the short circuit current when $V = 0$; point (ii) the knee point $\{I_{MAX}, V_{MAX}\}$ which is the point of maximum output power, $P_{MAX} = I_{MAX}.V_{MAX}$ and point (iii) $V = V_{OC}$ the open circuit voltage, when $I = 0$.

In the ZnO and chlorophyll based DSSC, upon shining light on the cell, the molecules of the chlorophyll dye absorbed on the ZnO nanofiboruos structure will be excited from their ground state to a higher energy state C to C* after absorbing photons ($h\nu$) as mentioned in the equation below:

$$\mathrm{hv} + \mathrm{C} \rightarrow \mathrm{C}^* \tag{2}$$

In their excited state dye molecule has higher energy content and overcomes the band gap of the semiconductor. The photo-excited chlorophyll dye molecules (C^*) will inject electrons into the conduction band of the ZnO and the excited dye will become oxidized or ionized (C^+). The reaction process involved is as follows:

$$\mathrm{C}^* + \mathrm{ZnO} \rightarrow \mathrm{C}^+ + \mathrm{e}_{\mathrm{cb}}^-(\mathrm{ZnO}) \tag{3}$$

The oxidized dye molecule is again regenerated by electron donation from the electrolyte (I^-) and the ground state of the dye (C) is restored and in turn iodide further oxidized to triiodide ions (I_3^-). The injected electron into the ZnO percolates through nanofiborous structure and is transported through an external circuit to a load where the work done is delivered as electrical energy. An electrical load can be powered if connected. The electron from the external load diffuses to the cathode where it gets transferred to the electrolyte, (I_3^-) so the electrolyte system is regenerated and the whole system is back to its original state to start a new cycle. These processes will continue as long as there is light and current is produced in the external circuit continuously [44].

In order to analyze the knee point, P_{MAX}, the expression for output power can be defined as;

$$P(V) = I(V)V = \left\{ I_{ph} - I_S \left[\exp\left(\frac{V}{V_T}\right) - 1 \right] \right\} V \tag{4}$$

The values of short-circuit current (I_{SC}) and open-circuit voltage (V_{OC}) of the assembled DSSC were found to be 20 μA, 0.321 V respectively. The maximum power density (P_{max}) of the DSSC as shown in Fig. 9b was found to be 0.176 μW/cm^2. An important parameter used to quantify the performance of the DSSC is the fill factor (FF) and is defined as;

$$FF = \frac{(I \times V)_{\max}}{(I_{SC} \times V_{OC})} \tag{5}$$

This dimensionless parameter is used to define the overall DSSC efficiency (η).

$$\eta = \frac{P_m}{E \times A_c} \tag{6}$$

Clearly, the quantities I_{sc}, V_{OC}, FF and η of a solar cell defined above are crucial performance characteristics. With the assembled DSSC, the fill factor (FF) was calculated as 44.02% and efficiency (η) as 0.13%. Further investigations on the degradation mechanism of solar cell and further testing techniques to improve the cell stability of this DSSC assembly are currently investigated in our laboratory.

4. Conclusion

To summarize, a novel of stainless steel mesh-based flexible quasi-solid dye-sensitized solar cell (DSSC) was designed and fabricated by using electrospun ZnO nanofibers web as photoelectrode and chlorophyll as the photosensitizer. The ZnO nanofibers fabricated by Electrospinning technique revealed the presence of highly porous fiber structure having ZnO grains with 12–20 nm size with high interconnectivity. The electrospun ZnO nanofibers based DSSCs exhibited good solar cell performance and based on the photovoltaic performance testing, an overall solar conversion efficiency of 0.13% was observed for the fabricated DSSC with a short-circuit photocurrent density of 28 μA, open-circuit voltage of 0.321 V and fill factor of 32.77%. This simple eco-friendly approach of fabrication of DSSC would help in the production and commercialization of low-cost solar cells for rural applications.

Acknowledgment

The authors are grateful to PSG & Sons Charities, Coimbatore for the facilities and support provided for carrying out this work.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary material related to this article can be found online at https://doi.org/10.1016/j.nanoso.2019.100311.

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Further reading

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