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Electrospun Nitrogen-doped TiO_2 Nanofibrous Thin Film for Photovoltaic Application

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Abstract*: This research study aims at fabrication of fine size nitrogen doped TiO_2 nanofiber using electrospinning method and evaluation of the performance of TiO_2 in a photovoltaic cell under visible light irradiation. Undoped and N doped TiO_2 nanoparticles were synthesized by sol gel method where titanium isopropoxide was used as the source of TiO_2 and ammonium nitrate was used as the source of N dopant. TiO_2/PVA composite material was prepared by stabilizing TiO_2 particle in to 10 wt % of PVA $_{(aq)}$ solution in order to prepare thin film that can be coated on photovoltaic (PV) cells. Coating of solid thin film PV cells by TiO_2/PVA nanofibers was conducted using electrospinning and doctor blade method. In both systems, doping the TiO_2 with nitrogen improved its optical properties which it successfully lowered the band gap energy from 3.14 to 2.76 eV and shifted its optical response to the visible light region. The presence of O-H stretching vibration, O-H bending and vibration of the N-Ti bond contributed to an increased performance of the PV cells. The electrospun N-doped TiO_2 produced better power output than doctor blade method coated PV cells with power of 0.040 and 0.026 mW, respectively.

Index Terms*: Electrospinning, Nitrogen doping, Photovoltaic, TiO_2.

I. INTRODUCTION

Semiconducting nanoparticles have emerged as an inevitable component in common energy sources due to their optical and catalytic properties, and it is enhancing alternative energy method to help on the world's rising energy demands [1]. Numerous researchers are looking forward in developing affordable, renewable and clean energy sources, and to reduce energy consumption as well as toxicity release to the environment with the aid of these nanoparticles [2]. Notably, TiO_2 nanoparticles have been intensively studied for its ability to photocatalytic and applications have high performance for photovoltaic application at cheap cost. It also shows low pollutant load, low toxicity, chemically stable and available at low price [3]. Although TiO_2 is recognized as an efficient semiconductor and photo catalytic material, the high energy band gap of TiO_2 limits its solar light absorbance capability and photovoltaic current generation [4],[5]. In order to overcome this issue by enhancing the performance of TiO_2 to absorb visible region solar light, several techniques such as sensitizing TiO_2 with dye and doping the TiO_2 with metal and non-metal element have been proposed [5], [6], [7], [8]. Among all these methods, doping TiO_2 by nitrogen (N) atoms has been studied intensively and N modification in TiO_2 is believed to narrow down the energy band gap efficiently and enlarge the solar light wavelength range [9]–[12].

Beside improving the chemical properties, increasing the surface area of TiO_2 film layer improve the photocatalytic efficiency by absorbing high amount of sensitized dye that lead high amount of light harvesting [13]. Also, high surface area of TiO_2 thin film allow electrolyte to diffuse through the cell intensively and results high photo electrochemical reaction rate. In addition, one dimensional nanostructure of TiO_2 thin film such as nanofibers increase the electron diffusion co efficient and results energy production to a great extent [14]. Considering these facts, TiO_2 thin film with highest surface area and high porosity can be constructed using TiO_2 – polymer composite nanofibers in order to increase the efficiency in photovoltaic applications. In this regard, electrospinning method has been proposed as a versatile method to synthesize metal nanoparticles incorporated nanofibers for various applications from biomedical field to energy applications.

Electrospinning is an advanced technique that produces continuous nanofibers from polymer solutions using electrical forces [15], [16]. By using this method, the physical properties of the polymers such as diameter, porosity, interconnectivity, orientation and compositions can be manipulated, and super thin films can be constructed with optimum parameters [16]-[18]. It is expected that these characteristics of electrospun thin films can aid to enhance the efficiency of energy generation. For instance, many studies have reported that metal nanoparticles incorporated electrospun fiber can be used as highly sensitive electrodes[19], [20], proton exchange membrane [21] and catalytic layers [22].

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In this work, the electrospinning technique is used to produce TiO_2 nanofibers with a high surface area that is capable to harvest a large amount of light and act as a sensitive electrode for PV cells. N doped TiO_2 and undoped TiO_2 incorporated PVA composite nanofiber thin film was produced using electrospinning method and highly efficient. Plant based natural dyes are recommended as an efficient and environmentally friendly source for absorbing visible dyes [23]. PV cell was constructed utilizing this modified TiO_2/PVA composite film. As a comparison, doctor blade method was used for coating and preparing of the PV cell.

II. MATERIALS AND METHOD

A. Synthesis Undoped and N-Doped TiO_2 Nanoparticles

First, N-doped and undoped TiO_2 nanoparticles were synthesized using sol-gel method. Titanium (IV) isopropoxide ($C_{12}H_{20}O_4Ti$, Sigma Aldrich) was used as the source of the TiO_2 and ammonium nitrate (NH_4NO_3, Sigma Aldrich) was chosen as the nitrogen source for N doped-TiO_2. During the sol-gel preparation, 6 ml of concentrated nitric acid (HNO_3) was added to 200 ml of distilled water. To prepare 10 wt% N doped TiO_2 suspension, 3.75g of NH_4NO_3 was added into the 40 ml of titanium (IV) isopropoxide and the resultant mixture was continuously stirred at 500 rpm for 1 hour at room temperature. For preparing undoped TiO_2 suspension, the same procedure was repeated without NH_4NO_3 dopant. Secondly, in order to prepare dry N doped TiO_2 and TiO_2 nanoparticles, obtained TiO_2 suspensions were poured into the crucible for drying and calcining. For the drying process, the suspension was dried at 90ºC for 3 hours in an oven. After that, the dried materials were calcined at 400ºC for 2 hours in air. The white crystal which consists of TiO_2 was obtained and the materials were then further ground into a powder by using pestle and mortar. White undoped and 10wt% N-doped TiO_2 powder was obtained.

B. Undoped and N-doped TiO_2 /PVA Polymer Solution

In order to prepare composite precursor solution, typical 10 wt% of polyvinyl alcohol (PVA) (M_w150000) (Sigma Aldrich) solution was prepared by dissolving 2g of PVA powder into 20 ml distilled water under vigorous stirring for 5 hours. Subsequently, 1 g of 10wt% N-doped TiO_2 powders were added into the 20 ml, 10 wt% of PVA aqueous solution in separate vials and all resultant mixture was continuously stirred for 30 mins.

C. Electrospinning of Undoped and N-Doped TiO_2/PVA Blended Solution

Electrospinning apparatus was set up by using 5 ml disposable plastic syringe equipped with a 23 G flat-end metal needle, syringe pump and high voltage power supply as depicted in Fig. 1. The blended solution TiO_2 – PVA precursor solution was transferred into the 5 ml disposable plastic syringe without air bubbles trapped into the syringe. At the counter electrode, an indium tin oxide coated (ITO) glass with a dimension of 2cm x 2cm glass was placed at the middle of the collector plate. The conductive side of the ITO glass was set to face the syringe pump in order to deposit electrospun fiber on the surface. The conductive side of the ITO glass can be tested by using a multimeter, where it should be continuity at the conductive glass surface. The counter electrode of the electrospinning set was earthed and the distance between the collector plate and the needle tips was fixed at 15cm. During the electrospinning process, 15 kV high voltage was applied to the metal capillary (needle) using a high voltage power supply. The syringe pump was set to feed the precursor solution with the constant flowrate of 2mL/h. Eventually, undoped and N-doped TiO_2 nanofiber were deposited on the ITO glass and electrospun fiber-coated glass was calcinated at 400ºC for 1 hour. After the calcination process, the ITO glass that was deposited with TiO_2 nanofiber were cooled down to the room temperature.

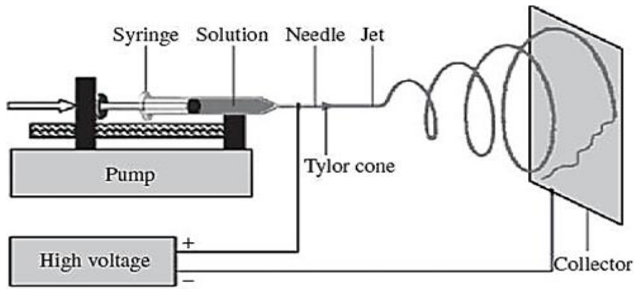


Fig. 1. Schematic diagram of electrospinning experimental setup for undoped and N-doped TiO_2/PVA fiber production

D. Doctor Blade Method Deposition of Undoped and N-Doped TiO_2/PVA Blended Solution

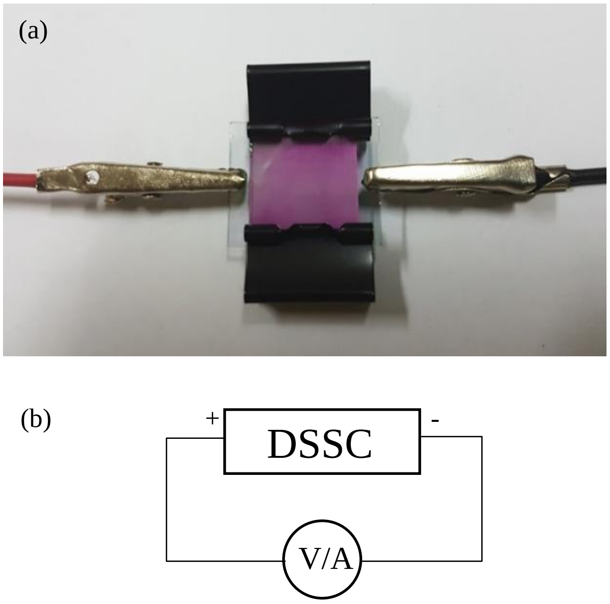
First, the ITO glass was taped at both the left and right side of the ITO glasses and undoped TiO_2/PVA blended paste was applied at the top edge of the ITO glass between the two pieces of tape. Then the blended paste was spread across the ITO glass using a glass rod. The gap between the strips of tape was filled with a layer of the blended solution. The same procedure was repeated with the N-doped TiO_2/PVA blended solution as well. After the undoped and N-doped TiO_2/PVA blended were deposited on the ITO glass by doctor blade method, the ITO glasses were calcinated at 400ºC using a furnace. After the calcination process, the ITO glass was cooled to the room temperature.

E. Staining of TiO_2 /PVA Thin Film and Photovoltaic Cell Assembly

Plant based natural dyes are recommended as an efficient and environmentally friendly source for absorbing visible dyes [23]. Anthocyanin pigment dye that was extracted from the flesh dragon fruit, was used for staining and sensitizing TiO_2 composite thin film in this study. Firstly, flesh dragon fruit was crushed to obtain juice and placed into the petri dish. The deposited surface of ITO glasses of undoped and N-doped TiO_2 /PVA were immersed into the dye for 10 minutes. During the soaking process, dye molecules were absorbed by the TiO_2 particles and the coated surface of ITO glasses was stained.

Photoelectrochemical cell preparation and photo electrochemical measurements were carried out as shown in Fig. 2. A carbon counter electrode was prepared by creating and coating the ITO glass with carbon soot under a candle. Afterward, dye sensitized TiO_2 coated electrode and carbon counter electrode were clamped together using paper clips. As a redox electrolyte solution, a small amount of anhydrous ethylene glycol dissolved potassium iodide/iodine solution was poured between the sandwiched electrode. Fig. 2 shows the diagram of the full assembly dye sensitized solar cell. The photovoltaic was then analyzed by a digital multimeter to obtain the data for PV cell performance.

A total of four samples of dye sensitized TiO_2 solar cells produced which consist of two methods, doctor blade method and electrospinning method. The performance of the photovoltaic cells produced in this experiment was determined by measuring the open circuit voltage, V_{oc}, and short circuit current density, J_{sc} by using digital multimeter. The photovoltaic cell was tested under the sunlight at three different times which was at 1.00 PM, 3.00 PM and 5.00 PM.



V = Voltmeter
A = Ammeter
DSSC = Dye Sensitize Solar Cell

Fig. 2. (a) Dye sensitized TiO_2 coated ITO glass electrodes, (b) dye sensitized TiO_2 PV cells for voltage and current measurements.

F. Characterization of the TiO_2/PVP Composite.

Morphologies of the electrospun TiO_2/PVP composite fibers were examined by Field Emission Scanning Electron Microscope (FE-SEM; Zeiss Supra 40). The samples were sputtered with gold.

UV-Visible Spectroscopy (UV-Vis; Agilent Cary 60 spectrometer) was used to study the changes in absorption wavelength for both undoped and N-doped TiO_2 samples. After electrospinning, the TiO_2/PVP composite fibers were pealed from the aluminum foil and characterized. UV-Vis absorbance spectra of all samples were recorded at a wavelength range from 200 nm to 800 nm.

Fourier Transform Infrared Spectroscopy (FTIR; Perkin Elmer) was used to study the changes in the functional group for the electrospun N-doped TiO_2 samples. After electrospinning, the deposited fibers were removed from the aluminum foils and tested. The scan spectra were recorded in the range from 500 cm^{-1} to 4000 cm^{-1}.

III. RESULTS AND DISCUSSION

A. Chemical Bonding and the Chemical State of Undoped and N-Doped TiO_2

The bonding characteristics of functional groups in N-doped TiO_2 samples and undoped TiO_2 sample were identified by FTIR. Fig. 3 shows the absorption peaks from FTIR spectra for the N-doped TiO_2 samples at two different concentrations, undoped sample and samples after the calcination process. The observed peak in the range of 3324.94 cm^{-1} -3369.00 cm^{-1} is attributed to O-H stretching vibration and the peak detected at 1634.70 cm^{-1} -1635.06 cm^{-1} is assigned to O-H bending. It is known that hydroxyl group plays an important role in the photocatalytic mechanism. Hydroxyl group was trapped by hole generated when TiO_2 was irradiated under UV light. Hydroxyl radicals formed suppress the recombination of electron-hole formation, hence increasing the TiO_2 photocatalytic efficiency [24]. The peaks around 1433.73 cm^{-1} and 1438.12 cm^{-1} are attributed to the vibration of the N-Ti bond [25]. There were few absorption peaks in the vicinities of 1700 cm^{-1} to 800 cm^{-1} from chelating of PVA with the titanium compound. After calcination at 400°C, the PVA absorption peaks were disappeared, indicating the decomposition of PVA.

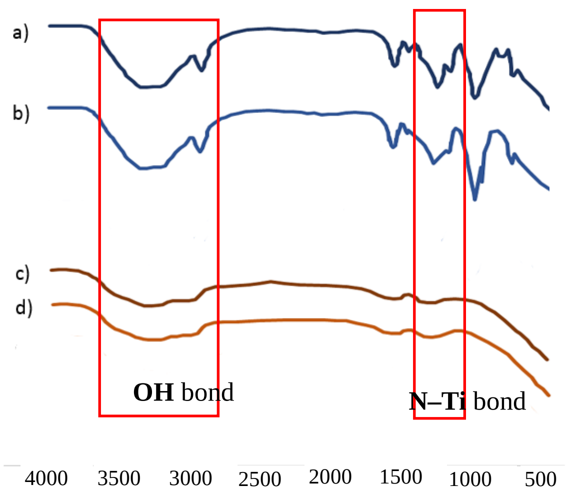


Fig. 3. FTIR spectra for (a) undoped titanium oxide, (b) N-doped titanium oxide, (d) undoped titanium oxide after calcination (c) N-doped titanium oxide after calcinations

B. Optical Properties and Band Gap Analysis of TiO_2 Thin Film Synthesized by Electrospinning Method

Undoped and N doped-TiO_2 thin films prepared by the electrospinning method were initially analyzed by UV-Vis technique in order to find out the absorption spectra and determine the band gap energy. The optical response for the undoped and N-doped TiO_2 samples were shown in Fig. 4. As seen in the Fig.4, the absorbance spectrum of N-doped TiO_2 sample significantly shift to the higher wavelength region and cut off wavelength of undoped TiO_2 is 395 nm whereas cutting off wavelength of N-doped TiO_2 increased to 450 nm. Absorption spectra shift to the higher wavelength of the visible region in the presence of dopant indicate that N-doped TiO_2 absorbs wider wavelength of light than the undoped TiO_2.

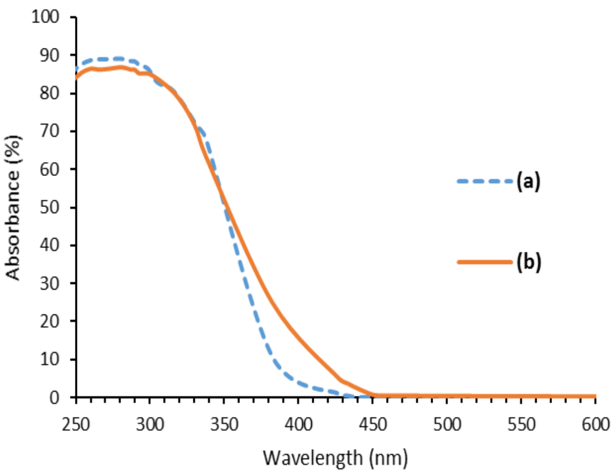


Fig. 4.UV-Vis absorption spectra of (a) undoped TiO_2 and (b) N-doped TiO_2

The relationship between the band gap energy and the cut off wavelength in absorption spectra was shown in equation (1) whereby band gap energy, E_{bg} (eV), plank constant, h=6.626 x 10^{-34} Js and speed light, c=3.0 x 10^{8} m/s:

$$E_{bg} = \frac{hc}{\lambda} \quad (1)$$

The energy band gap of TiO_2 electrospun membranes was determined using equation (1). Determined band gap energy along with the corresponding cut off absorbance wavelength are shown in Table I. Obtained band gap energy of the undoped TiO_2 sample was 3.14 eV. This value is consistence with the band gap range of TiO_2 that has been reported in the literature as 3.0 eV - 3.2 eV.

Table I. Band gap energy for undoped and N-doped TiO_2 samples

Sample	Cut-off wavelength (nm)	E_{bg} (J)	E_{bg} (eV)
undoped TiO_2	395	5.032 x 10^{-19}	3.14
N-doped TiO_2	450	4.417 x 10^{-19}	2.76

When nitrogen dopants were introduced in TiO_2, the band gap energy of N -TiO_2 reduced to a lower level which was 2.76 eV. This result indicates that N doping considerably narrows down the band gap energy of TiO_2. When TiO_2 is doped with N, N atoms distort the TiO_2 lattice and occupy the interstitial or substitutional position of the lattice [26], [27]. In this position, N 2p state overlap with O 2p and significantly rise the localized state of the oxide near to the top of the valency band or create a new inter band site that aid to pass the energy gap [26].

C. Morphology Analysis of Electrospun Undoped and N-doped TiO_2 / PVA Polymer Composite Fiber

FESEM micrographs of electrospun N-doped TiO_2/PVA and undoped TiO_2/PVA composite nanofibrous are shown in Fig. 5. Corresponding images of N-doped TiO_2/PVA (Fig. 5(a) and (b)) and undoped TiO_2/PVA (Fig. 5(c) and (d)) prove that continuous, discrete and randomly distributed TiO_2/PVA composite nanofiber has been produced by electrospinning technique. Furthermore, randomly elongated lumps appear on N-doped TiO_2/PVA composite fiber surface. It is speculated that these lumps ascribe to the dispersion of TiO_2 nanoparticle and these results illustrate that TiO_2 nanoparticles were consequently dispersed in PVA nanofiber [28].

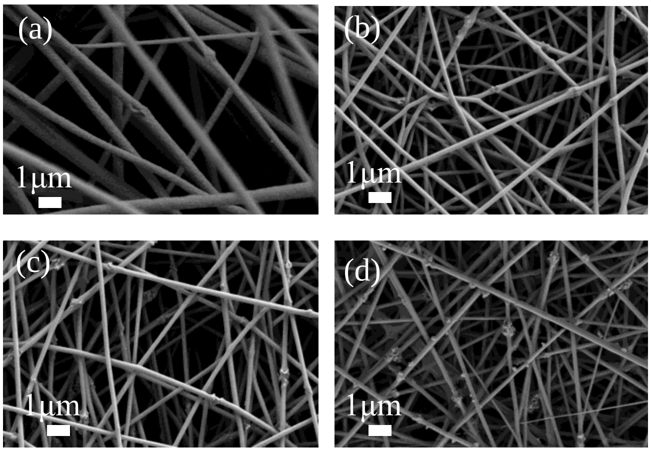


Fig. 5. FESEM images of undoped and N-doped TiO_2 electrospun fibers, (a) undoped TiO_2 fibers before calcination, (b) undoped TiO_2 fibers after calcination, (c) N-doped TiO_2 before calcination and (d) N-doped TiO_2 after calcination

Table 2 shows the mean diameter of undoped and N-doped TiO_2/PVA nanofibers. According to Table II, size of both N-doped TiO_2/PVA and undoped TiO_2/PVA were in the range of 200 nm to 700 nm which is the optimum size that offering a large surface area for the membrane [29]. However, the mean diameter of both calcined N-doped TiO_2/PVA and undoped TiO_2/PVA were 304±43 nm and 672±19 nm respectively whereas these values decreased to 202±72 nm and 342±38 nm after calcination. These changes in fiber diameter were obtained due to the evaporation of partial PVA and solvent molecules from the fiber during the calcination process [30],[31].

Therefore, it was expected that the higher rigid surface area and spaces between the fibers may harvest more solar energy and stimulate higher photovoltaic reaction than the continuous thin films.

Table II. Mean diameter of undoped and N doped TiO_2 fibers before and after calcination

Sample	Mean diameter before calcination	Mean diameter after calcination
Undoped TiO_2	672±19 nm	342±38 nm
N-doped TiO_2	304 ± 43 nm	202±72 nm

D. Photovoltaic Performance of Electrospun and Doctor Blade Method of Undoped and N-doped TiO_2 on Photovoltaic Application

Photovoltaic performances of doctor blade method of N-doped TiO_2 and undoped TiO_2 PV cells have been tabulated in Table III. Here in, produced circuit voltage (V_{oc}) and photo current density (J_{sc}) of each PV cells were recorded at different time period (1pm, 3pm and 5pm) of the same day in order to determine the average power output of doctor blade method and electrospun TiO_2 coated PV cells. Results show that the dye sensitized N-doped TiO_2 PV cells exhibit higher voltage and current at every time than the undoped TiO_2 PV cells. Overall, the average power produced by the N-doped TiO_2 PV cells (0.026 mW) was higher than the undoped TiO_2 PV cells (0.022 mW). These results are in agreement to the previous speculation which is N dopant has significantly increased the light absorbance efficiency of TiO_2 and enhanced photocatalytic performance of TiO_2 PV cells. Table IV shows the photo current density and voltage, generated by electrospun coated N-doped TiO_2 and undoped TiO_2 PV cells. Electrospun N-doped TiO_2 PV cells generated average 245.7 mV V_{OC} and 40.7 μAcm^{-2} J_{SC} whereas electrospun undoped TiO_2 generated 178.7 mV V_{oc} and 34.9 μAcm^{-2} J_{sc}. These observations comply with the previous finding that N-doped TiO_2 has efficiently absorbed wide range of light wavelengths and produced more energy than undoped TiO_2 [32],[33] . Table III and IV summarize that electrospun N-doped TiO_2 composite PV cells performed better than the doctor blade method. Generally, the higher value of V_{oc} and J_{sc} indicate the higher power output and better performance, which lead to a higher efficiency of photovoltaic cells [34]. In both N-doped TiO_2 and undoped samples electrospun coated PV cells produced 0.025 mW and 0.040 mW power respectfully whereas doctor blade method coated PV cells produced 0.022 mW and 0.026 mW power respectively.

Therefore, these results implicit that the electrospun TiO_2/PVA composite fiber has a great potential to produce photovoltaic current than TiO_2/PVA solid thin films. It is hypothesized that these higher power output of electrospun fibers obtained due to the high surface area of nanofibers that can harvest high amount of solar light and orientation of the fiber may improve the electrode mobility and charge transfer inside the cell [35]. In addition, the gap between the fibers (c.f Fig. 5) may trap the solar light efficiently tend to enhance the photovoltaic reaction.

Table III. Voltage and current density of doctor blade method coated titanium dioxide solar cells at different time interval

Time	1.00 PM		3.00 PM		5.00 PM		Average		Power
Sample	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**P (mW)**
Undoped TiO_2	158	35.7	153	35.6	149	35.6	**153.3**	**35.6**	**0.022**
N-doped TiO_2	179	38.0	169	37.9	166	37.8	**171.3**	**37.8**	**0.026**

Table IV. Voltage and current density of electrospun coated titanium dioxide solar cells at different time intervals

Time	1.00 PM		3.00 PM		5.00 PM		Average		Power
Sample	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**V_{oc} (mV)**	**J_{sc} (μAcm^{-2})**	**P (mW)**
Undoped TiO_2	184	35.2	177	34.8	175	34.7	**178.7**	**34.9**	**0.025**
N-doped TiO_2	250	41.3	248	40.6	239	40.3	**245.7**	**40.7**	**0.040**

IV. CONCLUSION

Undoped and N-doped TiO_2/PVA composite were deposited on ITO glasses by electrospun methods and doctor blade method. Initially, UV–Vis absorption spectra of undoped and N-doped TiO_2/PVA composite solution was analyzed.

Results indicated that N-doped TiO_2 sample significantly increase absorbance wavelength toward visible light region than undoped TiO_2. Consequently, the band gap energy of the N-doped TiO_2 lower than the undoped TiO_2 sample. These features considerably increase the photo absorption property of TiO_2. Based on the analysis performed, both electrospun and doctor blade methods coated samples for power output of N-doped TiO_2 was higher than undoped TiO_2 which indicate, the chemical modification of TiO_2 by Nitrogen doping enhanced the photo catalytic efficiency of TiO_2. Furthermore, the power output of electrospun coated PV cell was higher than the doctor blade coated PV cell. Higher output of electrospun fiber coated PV cells attributed to the higher surface area and electron exchange ability of TiO_2 /PVA nanofibers. Therefore, electrospinning technique has successfully improved the photocatalytic efficiency of N-doped TiO_2 nanofibers.

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