RESEARCH ARTICLE

Synthesis of TiO_2 nanorods with a microwave assisted solvothermal method and their application as dye-sensitized solar cells

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ABSTRACT

Inthiswork, Titanium dioxide (TiO_2) nanostructures have beensynthesized via a microwave assisted solvothermalmethod using titanium tetraisopropoxide (TTIP), polyvinylpyrrolidone(PVP) and Ascorbic Acid (AA) in ethanol. The mole ratio of PVP/AA was found to be critical in determining the morphology and crystal phase of the final product. PVP/AA mole ratio varied from 1 up to 15 to obtain different morphologies of TiO2. The structural analysis by XRD diffraction confirmed formation of titanium dioxide. The Williamson-Hall (W-H) analysis was used to study the individual contributionsofcrystallite sizes and lattice strain on the peak broadening of the TiO_2 nanoparticles.FTIR spectrum was used to estimate the various functional groups present in the nanostructures. Scanning electron microscope (SEM) images demonstrate nanoparticle, short nanorod, and long nanorods for 5,10 and 15 mole ratio of PVP/AA respectively. TiO2 nanoparticles and nanorodshave been used as photoelectrode in dye-synthesized solar cell (DSSCs) fabrication. The efficiencies of solar cells were calculated 3.23%and 4.01% for nanoparticles and nanorods, respectively.

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INTRODUCTION

Air pollution is one of the most important issues, which scientists face with it. Hence, many works have been done to reduce this problem in our surrounding environment. Using renewable energy such as solar energy instead of fossil fuel is one solution for the mentioned problem and photovoltaic cell (PV) [1-3] is a new technology that has attracted enormous interest recently because of inexhaustible, safe and environmentally friendly [4]. It is possible to find different categories of photovoltaic cells in the literature as silicon, copper indium selenite, CdTe,perovskite solar cell [5,6] and dye-sensitized solar cells [7-20].

Among the mentioned photovoltaic cells, Dye-sensitized solar cells (DSSCs) due to the high efficiency and low fabrication cost are more famous than that of the other ones. Moreover, two major requirements in DSSC technology are charge transport through a semiconductor and the electrolyte [4] which it can be possible to increase the electron transport and light trapping by using titanium dioxide (TiO_2) nanoparticle in DSSCs [21]. TiO_2 is used as an electron transport layer in PV andphotoelectrochemical devices and it has been implemented as a photocatalyst [22] and electrode solar cell based on dye-sensitized photo-electrochemical [23-29]. TheVarious kinds of TiO_2 structures like TiO_2 nanotubes [30] and one-dimensional TiO_2 [31-33] have been used to improve the efficiency of DSSCs.

From the fabrication point of view, different methods were used to fabricate TiO_2 nanostructures, which include these methods: Sol-Gel [34, 35],Micelle and Inverse Micelle, Hydrothermal, Solvothermal [36], Chemical Vapor Deposition, Electrodeposition, Sonochemical and

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Table 1. samples with different PVP to AA molar ratios

Sample name	(X= [PVP]/[AA])
S1	1
S2	5
S3	10
S4	15

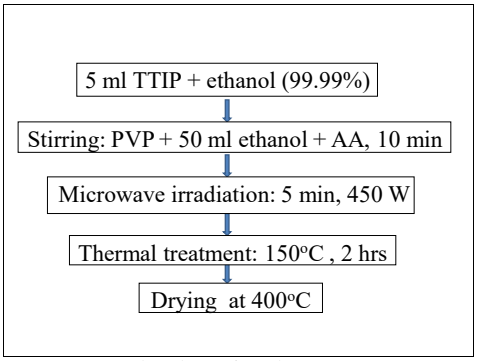

Fig.1. Flowchart of synthesize process.

Microwave [37-39]. Although, the Solvothermal method is more effective because, in this method, low temperature is used to format high particle crystal with high purity Using microwave helped solvothermal technique for quick heating and rapid crystallization rate [40, 41].

Titania nanotubes have been used vastly as a starting material compared to titanium dioxide since they have many hydroxyl groups and capability for ion absorption [42, 43]. Furthermore, it is possible to improve the electron transport as well as light trapping in DSSCs using TiO_2 nanowires and nanorods as scattering layer [44].

In this research, microwave assisted solvothermal method is used to prepare TiO_2nanorods. We introduce PVP as an effective capping agent for nanorods formation and ascorbic acid as a mild reducing agent in this report for the first time. TiO_2 nanoparticles and nanorodsare used as photoelectrode in dye-synthesized solar cell (DSSCs) fabrication.

EXPERIMENTAL METHOD

Preparing TiO_2 nanostructures

A mixture of Ti-containing precursor solution based on titanium tetraisopropoxide (TTIP)and polyvinylpyrrolidone(PVP) and Ascorbic Acid (AA) was preparedin 100 ml of ethanol with different molar ratio of TTIP/PVP/AA 1:1:X (X: 1, 5, 10, 15) as mentioned in Table1.Fig.1 shows Flowchart of synthesize process. For this solution, 5ml of TTIP was diluted in absolute (99.99%) ethanol.Thereafter, PVP was dissolved in 50 ml of ethanol and added to the first solution. Appropriate volume of AA was also dissolved in this solution and stirred for 10 min. Then the solution was exposed to microwave irradiation for 5 min at 450 W. The microwave treated solution transferred to a Teflon sealed autoclave for solvothermal synthesis and treated at 150°C for 2 h. The obtained powder was washed and calcined at 400°C to remove residual compounds and cooled naturally to room temperature for further analysis and cell fabrication.

Characterization tools

The crystalline structure of the powders was recorded by D8-Advanced BrukerX-ray diffractometer using Cu-Kα radiation (λ = 1.54056 Å) in the range 2θ = 20 – 90 degrees. SEM images were obtained using LEO 1450VP system. FTIR data were collected using an AVATAR-370-FTIR THERMONICOLET spectrometer using two separate procedures. The sample was unpacked into a tablet shape and put onto a polished silicon wafer before analysis.

Fabrication and characterization of DSSC

The TiO_2nanostructure wasmade by mixing with ethyl cellulose, α-terpineol and ethanol. The

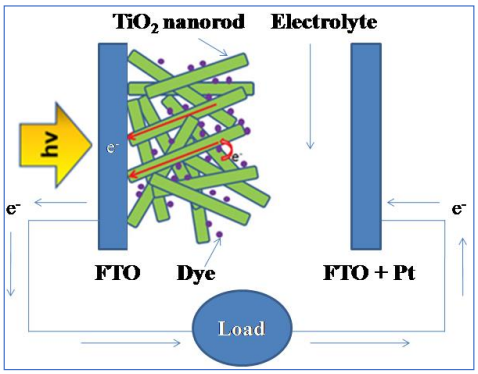

Fig.2. Schematic diagram of the cell structure.

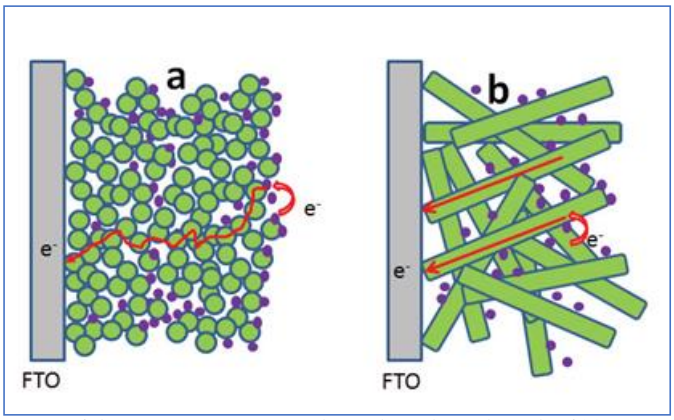

Fig.3. Schematic illustration of electron transportation in the working electrode made of (a) nanoparticles, (b) nanorods.

solution was stirred for 30 minutes. The solution sonicated together with heat treatment at 80°C until became to a viscous paste. A few drops of acetic acid and triton-x-100 added to the solution. The paste was spread on the FTO substrate by applying doctor blade technique. This is known as photoanode. The as prepared photoanode dried at 500°C for calcinations and sintering and finally the electrodewas treated in the solution of 40 mM $TiCl_4$ for 30 min. Next, the photoanode was soaked in 0.3 mMN719 dye for 24h. After that, the cells were filled with I^-/I^{-3}electrolyte. The counter electrode was Pt fabricated using thermal treatment of H2PtCl4 5mM at 400oC for 30 min.Figs. 2 and 3showthe schematic diagram of the cell structure.

Two main factorsdirectly affect the photovoltaic properties of a working electrode: surface area ofthe TiO2 layer and TiO2 crystal characteristic. The higher surface area would allowmore dye molecules to be absorbed on working electrode, hence generating more photoelectrons under the same level of excitation,while crystal property is important to electron transport. Electron transport within single crystals is fasterthan in a particle aggregate because the grain boundaries in the former are muchless. In our case, the TiO_2 nanoparticle layer had a very high surface area and dyeloading. A large amount of photoelectrons were generated and injected into thenanoparticles. However, the large number of grain boundaries at the nanoparticleinterfaces had caused a zigzag pathway of electron transport with ohmic loss. Charge recombination became a major obstacle in efficient energy conversion(Fig. 3a). The nanorods have higher surface area than the nanofibres but lower than the nanoparticles. However, as nanorods were in single-crystalline form, they could provide a better electron pathway for electron transport than nanoparticles (Fig. 3b).

The J-V characteristic of the cells having the

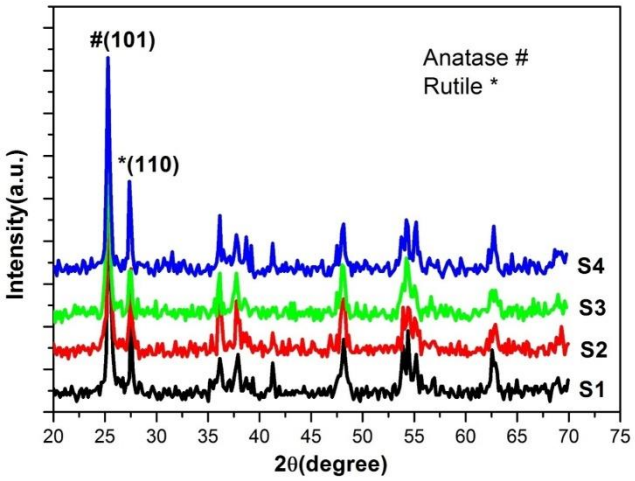

Fig.4. XRD pattern of the samples with different PVP/AA mole ratios.

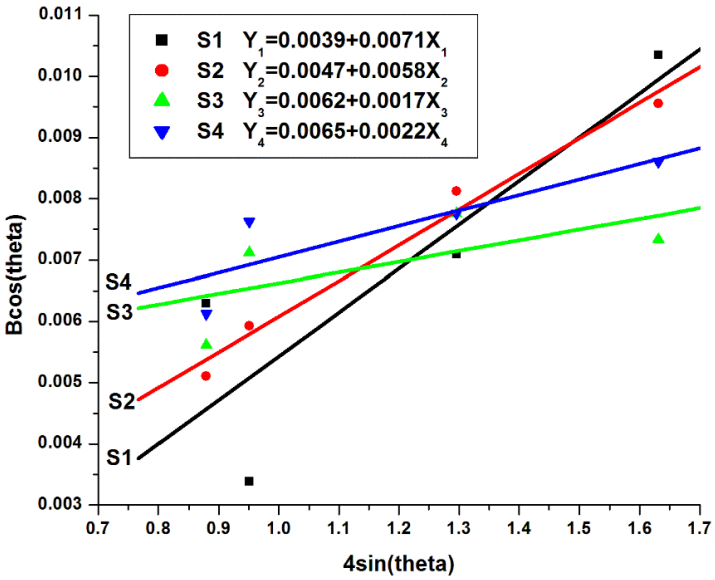

Fig.5. Williamson-Hall plot of samples for determination of lattice strains and mean particle sizes.

active area of 0.16 cm^2 was measured under AM 1.5 (100mWcm^{-2}) illuminations using a solar simulator coupled with a Palm SensPotentiostat for recording J-V plots. Incident photon-to-current efficiency (IPCE) was measured using a 150W halogen lamp in combination with a grating monochromator and calibrated by a silicon photodiode.

RESULTS AND DISCUSSION

Fig.4 shows that the prepared Titanium oxide nanostructures are well crystallized and composed of Anatase and Rutile phase structures. The (101)-plane for Anatase and (110)-plane for Rutile are the main diffraction planes seen in the figure. The net intensity for (101) shows that the samples are well crystallized. Increase of PVP/AA causes a considerable increase of the net intensity of the main peak of Anatase phase. This is the indication of preferential growth of Anatase phase due to the increase of PVP as capping agent. The mean crystalline size of nanoparticles is estimated using Williamson-Hall method [45, 46] and the results are plotted as Fig.5 and the calculated mean particle size and lattice strain are summed up in

Table 2. Williamson-Hall data of the samples

Sample name	0.9λ/d	Mean particle size	ε (%) (Lattice strain)
S1	0.0039	35.53	0.71
S2	0.0047	29.48	0.58
S3	0.0062	22.35	0.17
S4	0.0065	21.32	0.22

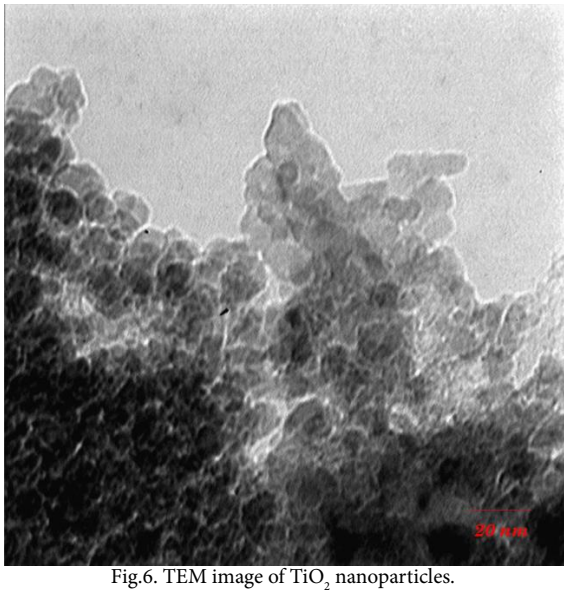

Fig.6. TEM image of TiO_2 nanoparticles.

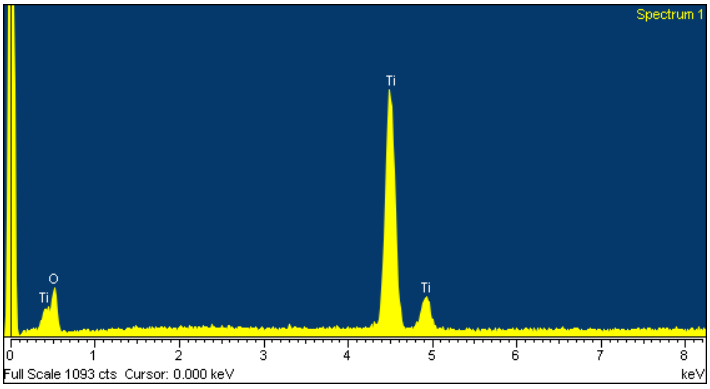

Fig.7. EDX spectrum image of TiO_2 nanoparticles.

Table 2. The mean particle size decreases from S1 to S4as well as the lattice strain accordingly. It is mostly stated that with the decrease of size, lattice strain increases unless the morphology of the particles changes dramatically [47, 48]. It can be stated that the increase of PVP/AA led to change of morphology of the samples.

Fig.6 shows TEM image of TiO_2 nanoparticles with a narrow size of about 20 nm which was in perfect agreement with the XRD analysis results. The Energy Dispersive X-ray Spectroscopy (EDS) confirms the chemical composition of the prepared TiO_2 nanoparticles (Fig.7).

Fig.8 indicates the FTIR spectra of the samples. There are absorption peaks for the wavenumbers of 2500-4000 cm^{-1}that is confirming the presence

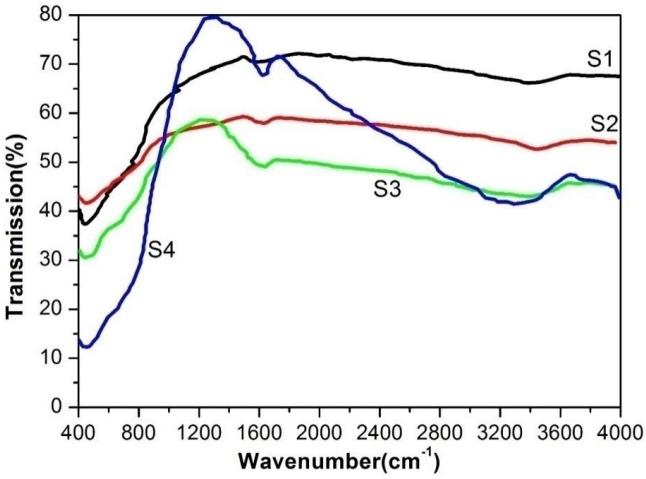

Fig.8. FTIR spectra of the samples.

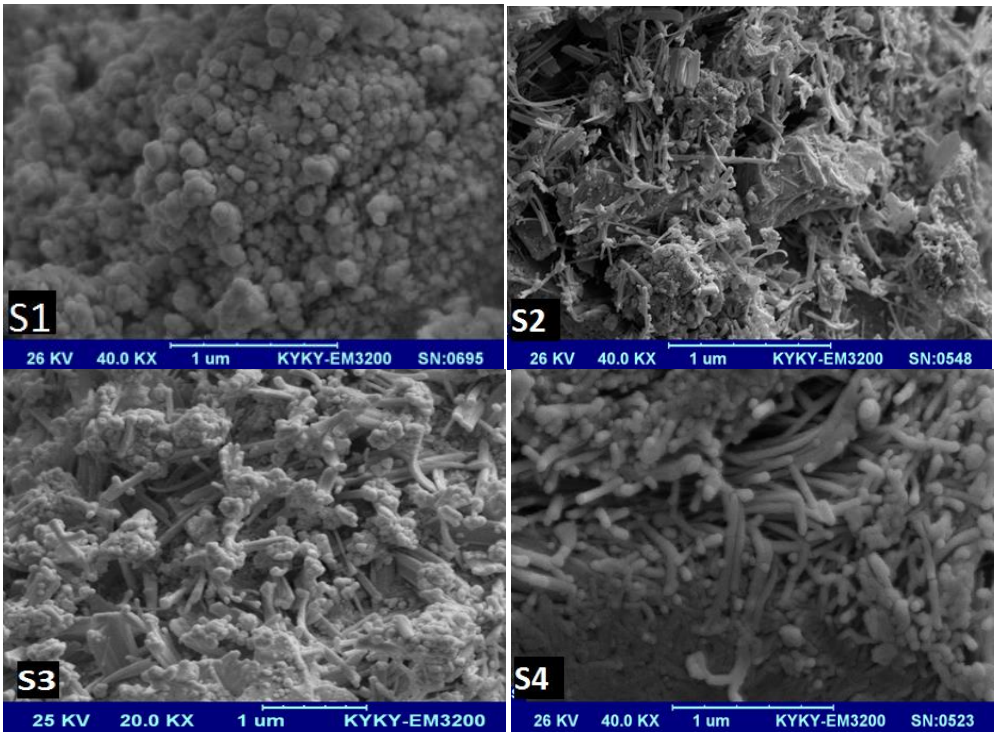

Fig.9. SEM images of the samples.

of compounds of carbon and water. The peaks at 1640 cm^{-1} in the spectra are due to the stretching and bending vibration of the -OH group. The peak at 440 $cm^{-1}$is attributed to Ti-O bond for S1, which shifts to 442, 450 and 454 $cm^{-1}$for S2, S3 and S4, respectively. The shift to higher frequencies indicates the shortening of Ti-O bond and simultaneously confirms the reduction of atomic plane distances.

Fig.9 shows SEM images for the samples. Generally, they have uniform size distribution which is the advantage of this method. Nanoparticles transform to nanowire like-structure when PVP/AA mole ratio increased up to 5 (Fig.9-S2). All nanowires are roughly uniform in shape and morphology in large area scale. Fig.9-S3 showed a uniformly long and narrow rod structure with several micrometers length

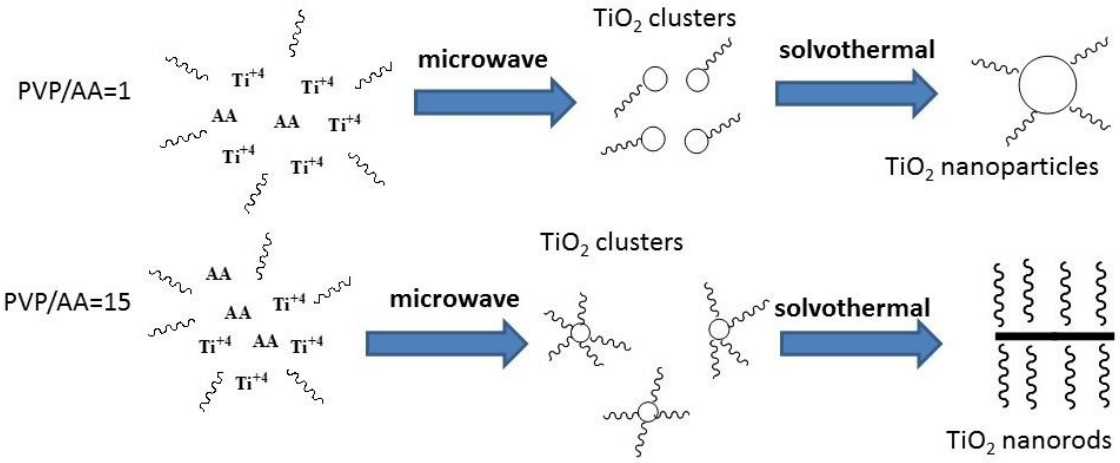

Fig.10. Schematic mechanism of the formation of TiO_2nanorods.

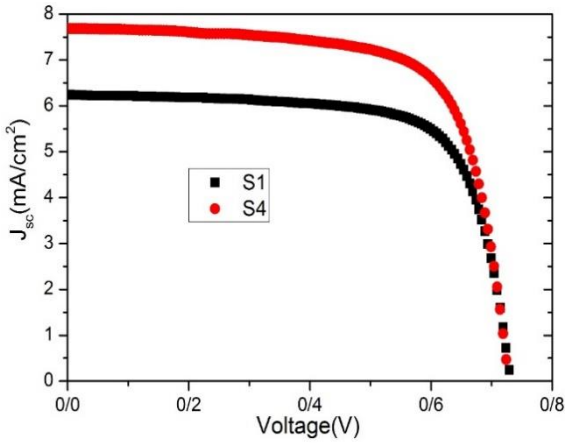

Fig.11. J-V plot of the cell fabricated using TiO_2 nanoparticles (S1) and nanorods (S4).

and 35 – 45 nm thickness. For S4 (Fig.9-S4) the colloidal solution is exposed by the excess of PVP and the morphology of nanostructures change dramatically to elongatednanorods. As the amount of PVP increased, the crystal facets of $TiO_2$are more influenced by PVP adherence. It means that PVP would be attached to the lateral planes with high surface energy and doesn'tallow the crystal to grow in that direction. It results to the formation of nanorods and nanowires. The more PVP is used, the better these facets are covered and thus longer and thinner they become (Fig.10).

The photocurrent density-voltage (J-V) and internal photocurrent efficiency (IPCE) characteristics of DSSC are depicted in Figs. 11 and 12, respectively. Samples with TiO_2 nanoparticles and nanorods used as photoelectrode under simulated air mass 1.5 global (AM 1.5G) full sunlight intensity. Detailed photovoltaic parameters, namely, open-circuit voltage (V_{OC}), short-circuit current density(J_{SC}), fill factor (FF), and the photovoltaic power conversion efficiency (η) have been obtained and tabulated in Table 3. J_{SC} is the parameter determined by the product of the charge carrier density under illumination, which shows the maximum number of the photo-generated carriers that can be extracted from a solar cell. The results demonstrated that the nominal values of Jsc were lower than the case of nanoparticle. The nominal efficiency of the prepared cell with nanorods was also lower than the nanoparticle case. The results indicate low

Table 3. Cell performance parameters extracted from J-V plots of nanoparticles and nanorods used as photoanode.

Cell	V_{oc} (v)	J_{sc}(mA/cm^2)	η(%)	FF (%)
S1	0.765	6.24	3.23	70.5
S4	0.765	7.5	4.01	72.1

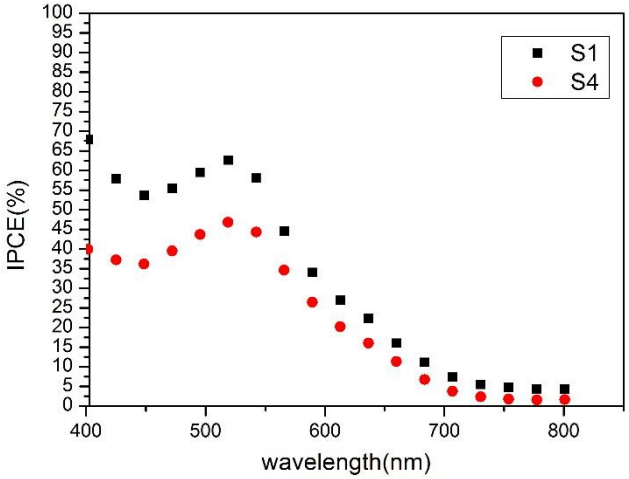

Fig.12. IPCE plot of the cell fabricated using TiO_2 nanoparticles (S1) and nanorods (S4).

photocurrent values compare to the reports for DSSCs [49-52]. IPCE values are indicating low values over visible to IR range which confirms low dye adsorption. This causes low performance of the solar cells. Although the IPCE of S4 is less than S1, the efficiency as well as J_{SC} of S4 is higher than S1 nanoparticle samples. This may be due to better morphology and TiO_2/organic interfacial interface and also could be the indication of the enhancement of electron transport rather than the particles which is confirmed also elsewhere [18]. It is also notable that high efficiency photoelectrode, in our research TiO_2nanorods sample, for DSSCs requires not only a high surface area for the loading of large amounts of dye molecules but also a closed net microstructure for light capture and facile electron transport [53].

CONCLUSIONS

Various TiO_2 nanostructureswere fabricated using a microwave assisted solvothermal method. PVP and ascorbic acid (AA) were used as a surfactant and reducing agents, respectively. The results show that PVP/AA mole ratio has a crucial effect on the morphology of final powder. XRD analysis showed that both Anantase and Rutile phases are available in the powders but with the increase of PVP/AA ratio, Anatase phase is only formed. Dye sensitized solar cell fabricated using TiO_2nanorods showed an efficiency enhancement due to the enhancement of short current circuit. It indicated that nanorods enhanced electron transport due to their preferential growth morphology.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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