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Effect of Binder Concentration and Dye Loading Time on Titania Based Photoanode in Dye Sensitized Solar Cell Application

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Abstract

The effect of binder concentration and dye loading time on the TiO_2/N3 based dye sensitized solar cell (DSSC) has been studied. Photoanodes for DSSCs were fabricated using the commercially purchased TiO_2 P25 (Degussa powder) nanoparticles and N3 dye. Two parameters are studied in this article. Firstly, the binder concentration affects the interconnectivity of TiO_2 nanoparticles. The other parameter studied is dye loading time. When the TiO_2 photoanode is exposed to a prolonged dye adsorption time, significant change is observed in the cell performance. The study correlates the change in open-circuit voltage (V_{OC}) and short-circuit density (J_{SC}) as the function of the dye adsorption time and reports the at-rest stability of the best-performing cell. The highest efficiency obtained in this study is 5%. The chronoamperometry study gives the stability of the generated photocurrent and the chronopotentiometry study determines the photovoltage stability of the device during the illumination period.

Keywords: Titania; N3 dye; DSSC; Artificial light.
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1. Introduction

Brian O'Regan and Michael Gratzel discovered an optically transparent film of 10 µm thickness with a high surface area which was coated with a layer of dye for light-harvesting application.[1] Dye sensitized solar cell (DSSC) consists of four major components such as mesoporous semiconductor material on conducting substrate, light-harvesting dye, electrolyte with redox couple, and a counter electrode as back contact.[2] The rate of electron transfer at semiconductor-dye-electrolyte junction affects the efficiency of DSSCs.[3,4] In a metal oxide, a large surface area is provided for the dye molecule to be adsorbed. When the light is incident, photogenerated electrons are injected into metal oxide and dye is regenerated by redox species present in the electrolyte.[5] The factor that reduces the efficiency is the binder concentration, dye loading time, and charge recombination which takes place in the semiconducting material/sensitizer and FTO/semiconducting material interfaces.[6–8]

Almalk *et al.* obtained an efficiency of 1.13% by using a modified ruthenium dye.[9] Wategaonkar *et al.* obtained an efficiency of 3.12% by using Nb-doped TiO_2.[10] Bhattacharya obtained an efficiency of 7.5% with the optimized dye loading time.[11] U. Mehmood obtained an efficiency of 4.03 % with 0.16% graphene-doped TiO_2.[12] T. Ganesh obtained an efficiency of 7.09% using carbozole (HMP) molecular dyes of high extinction co-efficient dyes.[13] Baglio obtained an efficiency of 1.4% using TiO_2-based DSSCs.[14] Kartini obtained an efficiency of 5.05 % with sensitized mesoporous TiO_2.[15] The fabrication of a nanocrystalline mesoporous titania photoelectrode is done following many methods. In general, nanocrystalline titania powder is made into a viscous suspension or paste by the addition of surfactants like terpineol or triton-X and filler material like ethyl cellulose. Surfactant reduces the surface tension among the nanoparticles to avoid aggregation. The filler material is used to create pores in the titania matrix. Usually, titania films are prepared by simple methods like doctor blading or screen-printing methods.

Here in the present research article, the dye sensitized solar cell is fabricated using TiO_2 photoanode sensitized with N3 dye and further sandwiched with the platinum counter. The influence of binder concentration and dye loading time was studied by recording its J-V characteristics.

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Chronoamperometry and chronopotentiometry were carried out to study the short-circuit current and open-circuit voltage of various dye-loaded films.

2. Experimentals

All the materials used in the fabrication and synthesis were of analytical grade. They were used as purchases including TiO_2 (P25 Degussa Nanoshel LLC, US), FTO glass ((FTO- Sigma Aldrich, USA Ethanol (Changshu Hongsheng Free Chemical Co. Ltd.), ethyl cellulose (SDFCL, India), Terpineol (KPS Ltd., India), acetylacetone (HPCL, India), and N3 dye (Sigma Aldrich)

2.1 Fabrication of TiO_2 paste using different binder concentrations

Figure 1 shows the schematic representation of the preparation process of TiO_2 paste. TiO_2 paste was prepared using P25 Degussa powders, ethyl cellulose, and acetylacetone. The paste was prepared by the previously reported method.[16-18] Briefly, the paste was prepared by mixing TiO_2 nanoparticles, ethyl cellulose (0.2, 0.3, and 0.4 g), terpineol, and acetylacetone in ethanol. The paste with different concentrations of ethyl cellulose was evenly spread on the FTO using the doctor blade technique. The photoanodes were then dried and annealed at 450 ℃ for one hour. Fig. 1a shows the doctor blade technique for the fabrication of photoanode.

2.2 Sensitization of N3 dye on TiO_2 film

0.3 mM N3 dye solution was dissolved in ethanol. Three different TiO_2 photoanodes named Cell 1, Cell 2, and Cell 3 were dipped in N3 dye at various dye loading time intervals of 15, and 30. 60. 90, and 120 min. The sensitized photoanodes at various dye loading times were further used for DSSC application. Fig. 1b shows the growth mechanism of N3 dye onto TiO_2. Fig. 1c shows the photograph of TiO_2 photoanode for various dye loading time intervals.

2.3 Preparation of polyiodide electrolyte

Polyiodide solution was used as a liquid electrolyte consisting of 0.5 M of lithium iodide (SRL), 0.05 M of iodine (Fisher), and 0.15 M tertiary butyl pyridine (ACROS organics) in 100 mL acetonitrile (s-d fine chem. Ltd). The mixture of the above precursor's solutions was kept under magnetic stirring for 30 min at ambient conditions till all the precursors got completely dissolved.

2.4 Solar cell assembly

A working electrode with an active area of 0.25 × 0.25 mm was used with a 45 μm spacer thickness at the edge. The counter electrode of platinum facing towards the TiO_2 photoelectrode was held together using binder clips on the opposite end. A drop of polyiodide electrolyte was dropped between them till no air bubble was formed. N3 sensitized TiO_2 cell so formed was characterized by its photovoltaic performance.

3. Characterization techniques

The crystallite size of TiO_2 film onto FTO was determined using X-ray Diffraction (XRD) (model: XRD, Rigaku "D/B max-2400", Cu Ka = 0.154 nm). The morphology and

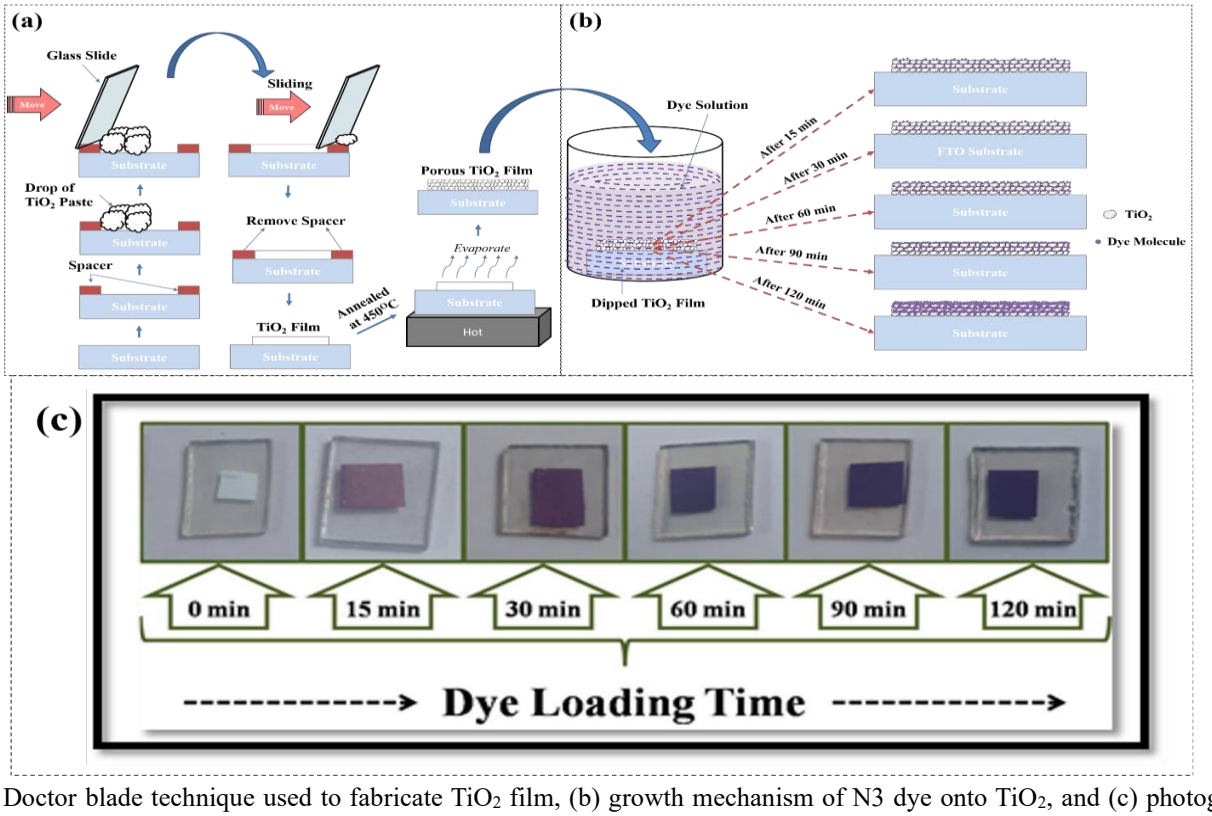

Fig. 1 (a) Doctor blade technique used to fabricate TiO_2 film, (b) growth mechanism of N3 dye onto TiO_2, and (c) photograph of TiO_2 photoanode sensitized with N3 dye for various dye loading times.

cross-section images of TiO_2 film and N3 dye adsorption on TiO_2 film were determined using scanning electron microscopy (model: JEOL-JSM 6360-A). UV–Visible spectrophotometer (model: JASCO V-670) was used to test the optical properties. Photovoltaic parameters of each cell were measured using a simulated solar stimulator (Oriel Sol 2 A, Newport). LED with 35 mW/cm^2 and 2420 Keithley source meter was used to measure J-V characteristics. Chronoamperometry and chronopotentiometry were measured using potentiostat/galvanostat (IVIUM vertex model).

4. Results and discussion

4.1 X-Ray Diffraction: Structural properties

The films fabricated were characterized for crystal size and phase by X-ray diffraction. Fig. 2 shows the XRD patterns of various films fabricated by various binder concentrations. The binder concentration is varied from (0.2-0.4 gm). Anatase and rutile phase is confirmed using JCPDF no. 21-1272 and 21-1276, respectively. (101), (004), (200), (105), (211), (204), (220), and (301) orientations were observed. The Scherrer formula[19–21] was used to calculate the average crystallite size of TiO_2 photoanode which was observed to be nearly equal to 25 nm. Smaller crystal size provides a large surface area which is useful for dye adsorption. It is previously reported that a single crystalline phase performs better than a mixed phase.[22] Porosity is an important factor in the dye adsorption phenomenon. As the porosity increases, the dye adsorption capacity increases Fig. 3a represents the 2D image of TiO_2, and Fig. 3b represents the 3D image of TiO_2. The Ti^{4+} atoms are shown in blue and O^{2-} is shown in red. Here, TiO_2 exhibits an anatase phase with $\alpha = \beta = \gamma = 90°$ and lattice parameters a = 3.80271Å, b = 3.80271Å, and c = 9.74775Å. The unit cell volume is equal to 140.958382 Å^3. The schematic representation of the crystal structure of TiO_2 is done using VESTA 3 used for three-dimensional visualization of crystal, volumetric, and morphology data.[23]

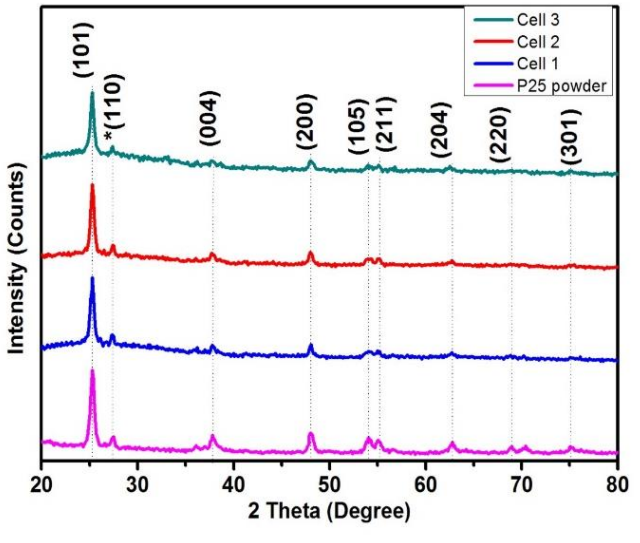

Fig. 2 XRD pattern of TiO_2 film fabricated using various binder concentrations.

4.2 UV-visible spectroscopy: optical studies

Figure 4 shows the optical absorbance spectra of photoanodes fabricated using various binder concentrations. The energy bandgap is observed to be 3.2 eV, which is determined by extrapolating the linear line portions to the energy axis.[20,24] Figs. 5(a-c) shows the optical absorption of TiO_2 photoanodes for various dye loading times varied from 15 to 120 min. Here, the effect of N3 dye loading onto TiO_2 photoanode is observed. The dye loading absorption is observed in the range of 425-650 nm. Here, we observed that absorption is the highest for 90 min dye loading period. The adsorption shows a redshift and is broadened with a prolonged dye loading time.[2,25,26] It is observed that after 90 min dye loading time, the adsorption is decreased due to the aggregation of dye molecules.

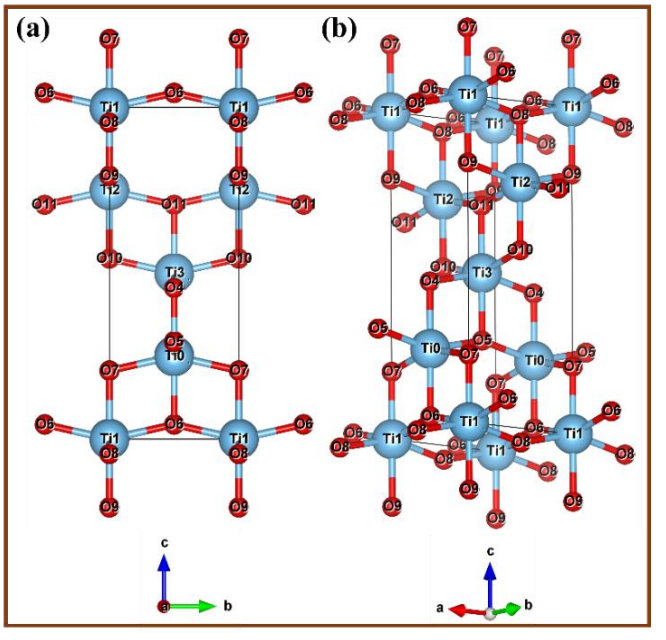

Fig. 3 (a) 2D image, and (3D) image of TiO_2 crystal structure using VESTA. Reproduced with the permission form[23], Copyright International Union of Crystallography.

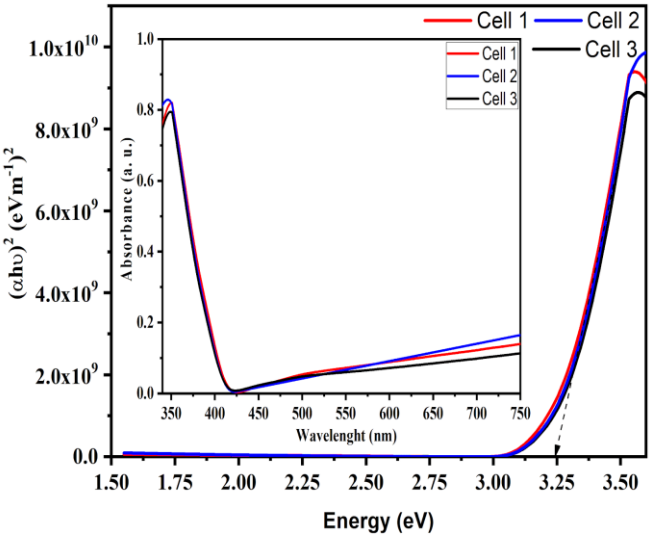

Fig. 4 Optical absorption spectra (in inset) and Tauc plot of the TiO_2 films.

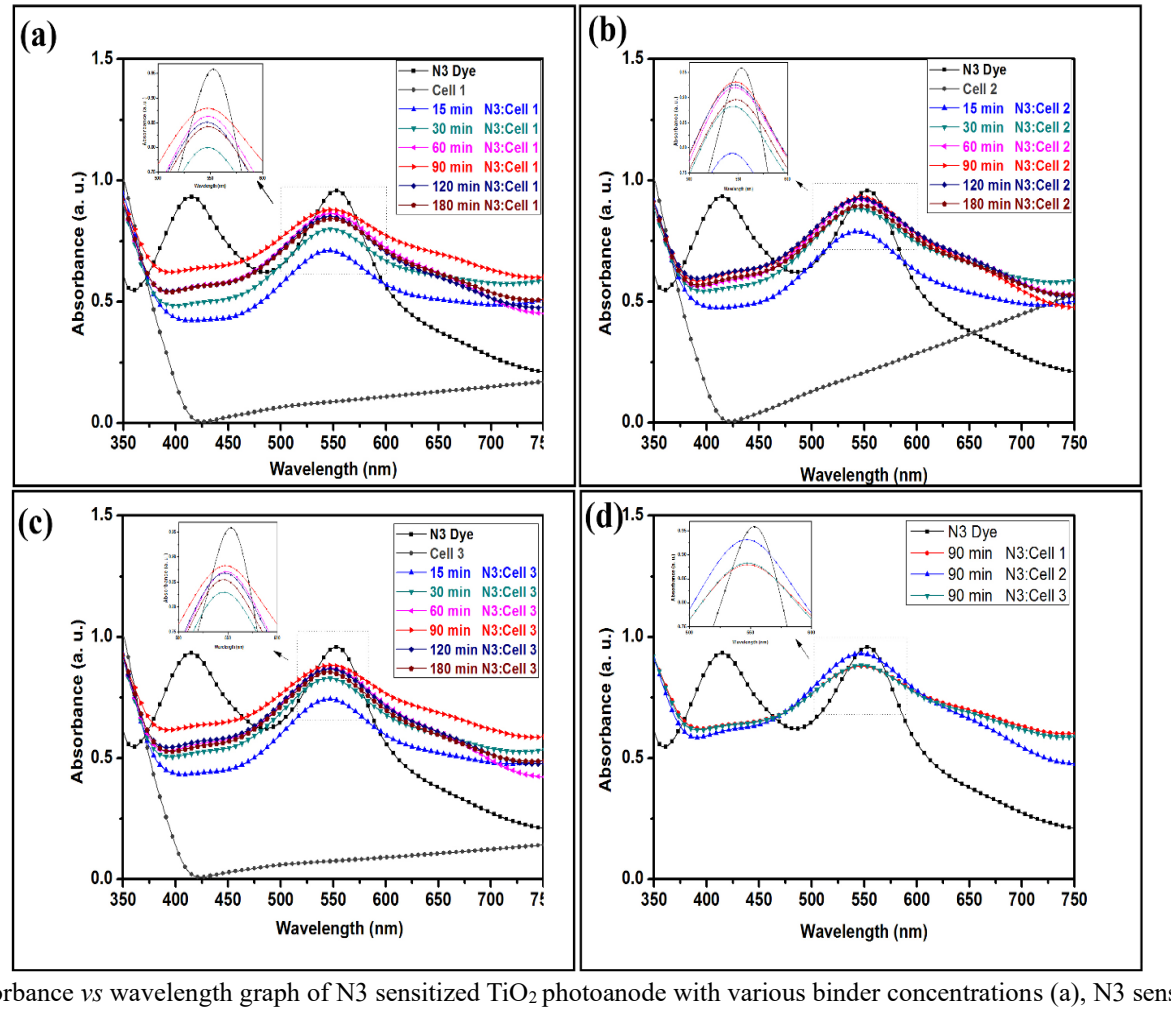

Fig. 5 Absorbance *vs* wavelength graph of N3 sensitized TiO_2 photoanode with various binder concentrations (a), N3 sensitized cell 1 (b), N3 sensitized cell 2 (c), N3 sensitized cell 3 (d) optimized 90 min N3 sensitized Cell 1, Cell 2, Cell 3.

4.3 Scanning electron microscopy: morphological studies

Morphology is affected by various binder concentrations varied from 0.2– 0.4 gm binder concentration. Fig. 6 shows the increasingly porous nature with an increase in the binder concentration. At lower binder concentrations, the films are densely packed with cracks. The effect of densely packed films is such that the dye adsorption capacity is low as the porosity is less. At higher binder concentrations, the films are very thick and take a long time to be dried. The effect is such that there is no interconnectivity between the TiO_2 as they are much more porous than needed. The fabricated cell thus further leads to lower efficiency of the cell. Figs. 7a and 7b show the SEM image of cell 2 at various magnifications, respectively. The films show porous morphology required to study the effect of dye loading time. The SEM images show that the films tend to observe N3 dye. Fig. 7c shows a cross-section image of TiO_2 cell 2. The cross-sections give the thickness of films approximately equal to 25-30 µm.

4.4 J-V Characteristics

Figure 8a shows a schematic representation of the N3-sensitized TiO_2 photoanode. The working principle of DSSC is explained such that when light i.e. photons are incident on the solar cell, the electrons from the dye get excited from the highest occupied molecular orbitals (HOMO) to the lowest unoccupied molecular orbital (LUMO). The electrons present in the LUMO of N3 get injected into the conduction band of the wide band gap semiconductor, which results in the de-excitation of the dye. The excited electrons from the conduction band of the semiconductor move to the conducting transparent substrate (TCO) connected to an external circuit. The current flow through the circuit and light energy are converted into electrical energy. To regenerate electrons in the HOMO of dye, liquid electrolyte plays an important role. Polyiodide electrolyte containing I^-/ I_3^- redox couple ions is used as an electron mediator between the semiconducting photoelectrode and counter electrode. Therefore, the de-excite quantum dots (photosensitizer) are regenerated by receiving electrons from the I^- ion redox mediator that get oxidized to I_3^-. In this process, the reduction of electrolyte occurs. The I_3^- substitutes the internally donated electrons with that from the external load and is reduced back to the I^- ion. The movement of electrons occurs due to the diffusion of charge compensating cations in the electrolyte layer in the conduction band of the wide band gap nanostructured semiconductor. Therefore, the generation of electric power in the DSSC

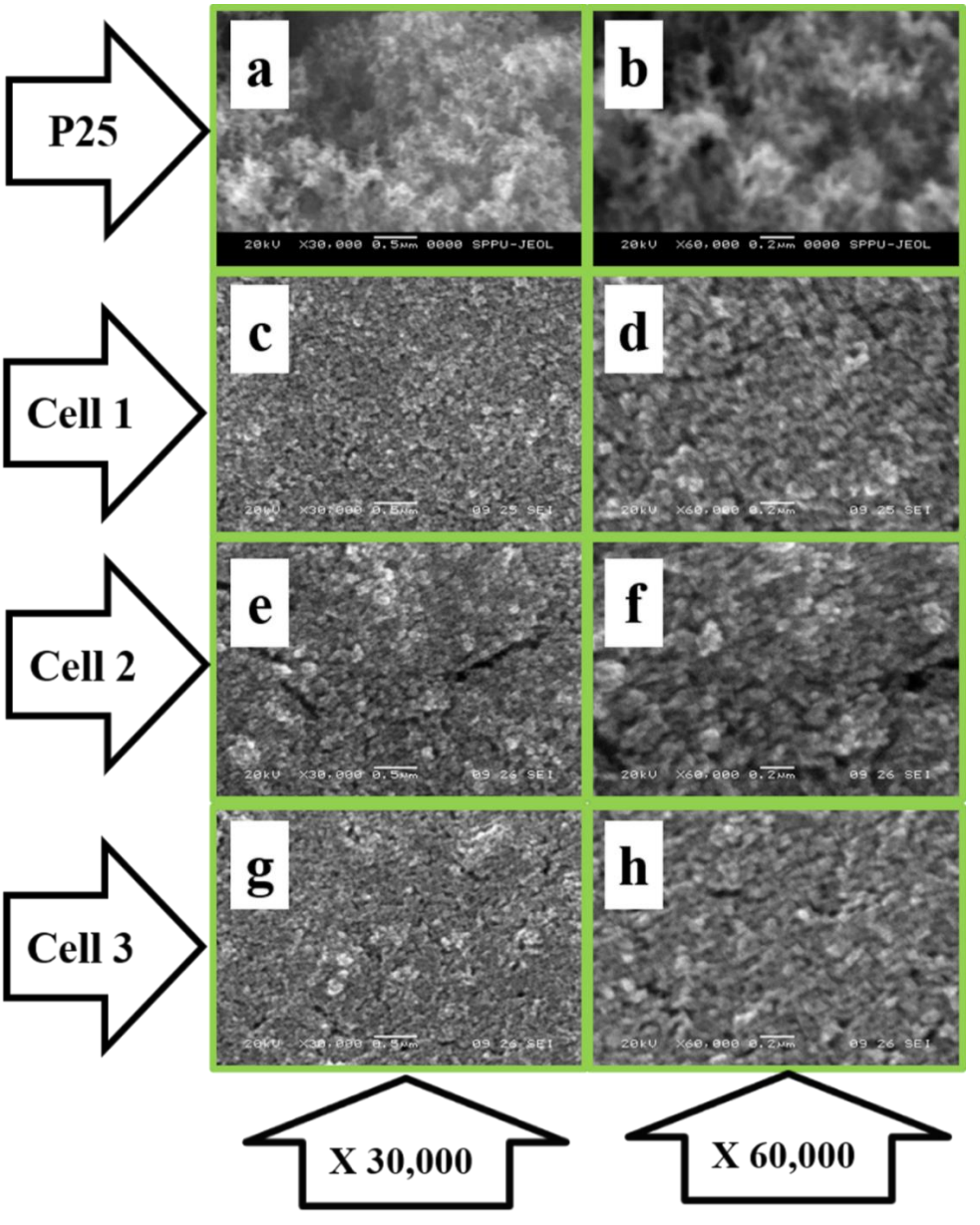

Fig. 6 SEM micrographs of TiO_2 P25 powder and prepared photoanodes with various binder concentrations (a, b) TiO_2 P25 Powder, (c, d) (0.2 gm) TiO_2 Cell 1, (e, f) (0.3 gm) TiO_2 Cell 2, (g, h) (0.4 gm) TiO_2 Cell 3.

causes no permanent chemical change or transformation. Fig. 8b shows the J-V characteristics of TiO_2-sensitized N3 cell 2. By using *J-V* characteristics, various parameters are studied. When the *J-V* characteristics are studied, it is observed that the efficiency goes on increasing up to 90 min dye loading time. After 90 min dye loading time, it is observed that the photocurrent density is decreased. The overall efficiency increases from 0.36% to 5.37%. Afterward, the efficiency decreased up to 4.43%. The reason for the decrease in efficiency is due to the aggregation of dye molecules at the TiO_2 photoanode. U Mehmood *et al.*[27] reported 0.6% efficiency for TiO_2 multiwall carbon nanotube. Ogunsolo *et al.*[28] reported 4.2% efficiency and studied the recombination events in DSSCs. Horiuchi *et al.*[29] reported 7% efficiency based on metal-free indoline dyes. Zhang *et al.*[30] reported 1.8%concerning the time of the fabricated device. Similarly, efficiency using flexible substrates. Chen *et al.*[31] reported 8.4%chronopotentiometry is the built-in voltage study with time efficiency with the enhanced light-absorbing property of ruthenium complexes for DSSCs. Song *et al.*[32] reported 5.0% efficiency for DSSCs. Chau *et al.* reported 5.1 % efficiency and studied particle size effect on the fabricated DSSCs. Chao-Po Hsua *et al.*[33] reported 6.1 % efficiency for low-temperature fabrication of TiO_2. Bandaranayake *et al.*[34] reported 8.2% efficiency. Lee *et al.*[35] reported 5.3 % efficiency and studied the surface morphology. Patrocínio *et al.*[36] reported 5.3 % efficiency for TiO_2-based DSSCs. Liu *et al.*[37] reported 7.4 % efficiency when tantalum was doped in TiO_2 film. Pan *et al.*[38] reported 2.2 % efficiency with TiO_2 nanoribbons and nanorods.

4.5 Chronoamperometry and chronopotentiometry

Chronoamperometry is the study of built-in current

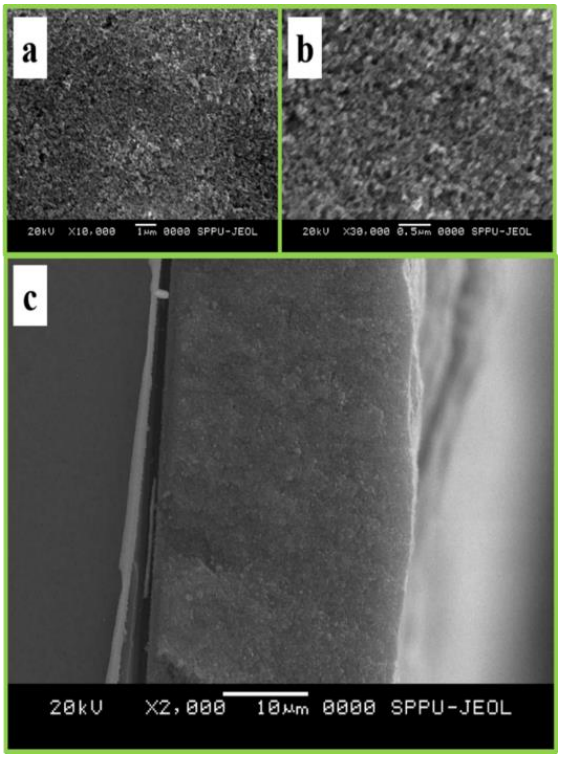

Fig. 7 (a, b) surface morphology and (c) cross-section SEM micrograph of cell 2.

intervals. When light is incident on the fabricated cell, the photocurrent or photovoltage is generated and the maximum generated photocurrent and photovoltage is observed for time intervals of 10 sec on and 10 sec off cycle. Using this characteristic measurement, the current and voltage stability of the fabricated device is studied. Here, chronoamperometry and chronopotentiometry are studied using the same cell used in the characterization of J-V characteristics. Fig. 9a shows the open-circuit voltage (V_{OC}) for the repeated ON/OFF incident light periods for various dye loading time intervals. As the light is incident on the cell, the photovoltage increases with an increase in dye loading time for 100 seconds. The photovoltage increases from 15 min dye loading time to 90 min dye loading time. Here, we can see that the results are following the JV characteristics. The V_{OC} increases from 0.49 to 0.66 V. After 90 min dye loading time, the V_{OC} decreases up to 0.59 V. Fig. 9b shows the current *vs* time graph of N3 sensitized TiO_2 cell 2 for various dye loading time intervals at light ON-OFF irradiation conditions. Here, we can see the effect of dye loading time on the catalytic activity of the photoanode. The current is observed to be stable for up to 100 secs. This current show the photoelectrochemical stability of the PA/electrolyte interface in all the cells. Here, the results are following the J-V characteristics. It is observed that the

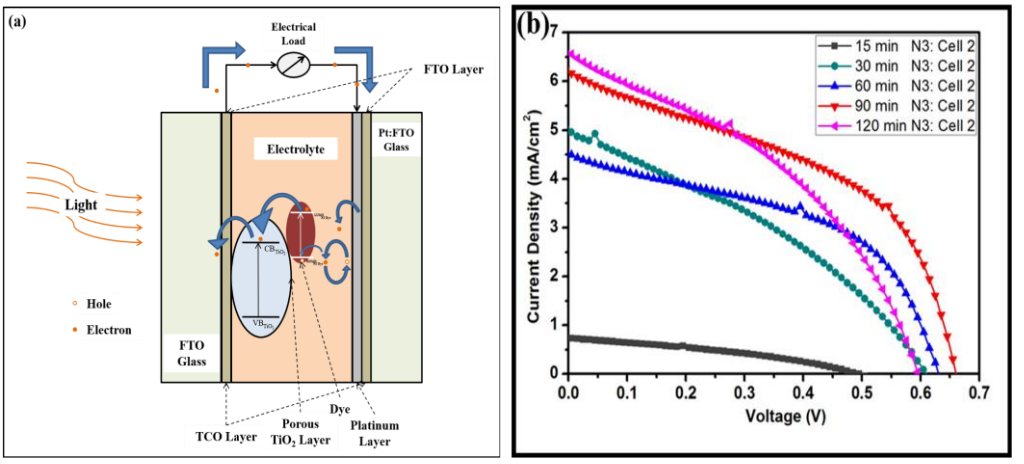

Fig. 8 (a) Schematic representation of the working principle of DSSC and (b) *J-V* characteristic graph of N3 sensitized TiO_2 photoanode for various dye loading films.

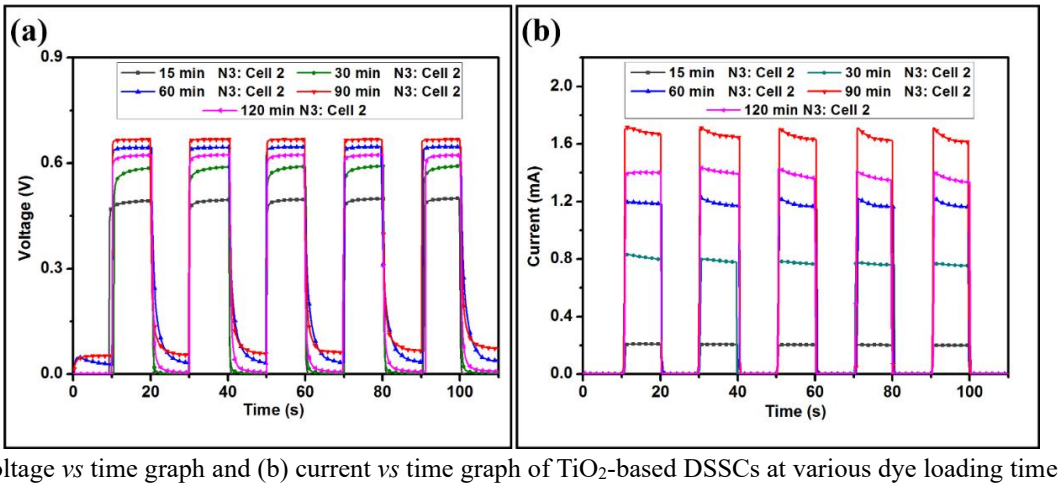

Fig. 9 (a) Voltage *vs* time graph and (b) current *vs* time graph of TiO_2-based DSSCs at various dye loading time intervals.

current increases from 0.74 to 6.2 mA/cm^2 and then decreases up to 6.61 mA/cm^2. All the results of V_{OC} and current density are in agreement with the J-V characteristics.

5. Conclusion

The effect of dye loading time on the titania photoanode is systematically investigated. Also, the article has brought out the study of binder concentration and the effect of the adsorption of dye. The result shows a better performance for 0.3 g of binder concentration and 90 min dye loading time. For prolonged dye loading time, the performance of the cell decreases due to dye aggregation. The morphology shows a porous nature, which is helpful for the dye adsorption phenomenon. This TiO_2-sensitized N3 dye clamped with a counter electrode using a polyiodide electrolyte-based dye sensitized solar cell is fabricated with proper optimization of dye loading time. Efforts are being carried out to study other parameters affecting the DSSCs.

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Conflict of interest

There are no conflicts to declare.

Supporting information

Not applicable.

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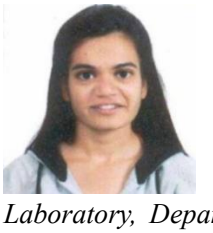

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