Effect of Dye Absorption Time on the Performance of a Novel 2-HNDBA Sensitized ZnO Photo anode Based Dye-Sensitized Solar Cell

Shital S. Rakhunde,[1] Kisan M. Gadave,[1,*] Dnyaneshwar R. Shinde[1] and Pankaj K. Bhujbal[2]

Abstract

In the present study, dye-sensitized solar cells (DSSCs) were fabricated using novel [(2-hydroxy-1-naphthyl) diazenyl] benzoic acid (2-HNDBA) dye. The effect of dye absorption time on the performance of DSSCs was studied. The doctor blade method was employed to prepare the ZnO seed layer. The structural, morphological and optical properties of the ZnO photoanode were studied systematically. The syntesized ZnO was found to be of hexagonal wurtzite structure. The 2-HNDBA sensitized zinc oxide based DSSCs have been studied for its photoresponse at the dye absorption time from 60 -240 min. The fabricated DSSC prepread at 60 min dye absorbtion time was found to exibit a fill factor and a photo conversion efficiency of 0.44 and 1.09 %, respectively. As the preparing time increases to 120 min, the respective parameters increase to 0.53 and 2.08 % at 120 min. However, on further increasing the dye absorbtion time, the fill factor and photo conversion efficiency are reduced to 0.51, and 1.69%. Thus, 120 min dye absorbtion time has been found to be the optimized dye absorption time for novel 2-HNDBA dye-sensitized ZnO photoanode based DSSCs.

1. Introduction

Nowadays, the energy demand is rising in the world from the last four decades and reaches up to the rate growth at 1.8% per year.[1] Generally, energy is divided into renewable and non-renewable resources.[2] Renewable energy is energy generated from sources like ocean, hydropower, wind, biomass, geothermal resources, hydrogen, and solar energy.[1] Solar energy is directly used as heat energy or conversion from sunlight into electricity directly as well as indirectly.[3,4] The sun is approximately 3×10^{24} J per year radiation, which is ten times current energy demand.[5,6] Solar electricity is produced from the conversion of solar radiation into direct current electricity using semiconductors in photovoltaic devices by photovoltaic effects.[3] It produced clean, cheap, safe, high efficiency, and good stability.[7] Photovoltaic techniques are divided into first, second and third generation.[8] The first third-generation DSSCs were reported in 1991 by Gratzel *et*

al.[9] The current thin films solar cell and crystalline and amorphous silicon solar cells are used to convert solar energy into electricity. However, there are some restrictions such as toxicity, high production time, production cost, and types of substrates used for the fabrication device. This has attracted researchers to find its alternatives. A dye-sensitized solar cell (DSSC) is the third generation solar cell. It is one of the most promising solar cell devise than crystalline solar cells and thin-film solar cell devices due to their simple manufacturing, high power conversion efficiency, low production cost, and non-toxicity.[10] In DSSCs, metal oxide semiconductor has good stability under irradiation in liquid solution but it cannot absorb visible light due to its wide-bandgap. If these types of materials are sensitized with a photosensitizer such as dyes then they work as photo-anode in DSSC's. The photosensitizer in the adsorbed form on the metal oxide undergoes electronic excitation on irradiation with visible radiations. The excited state electron from lowest unoccupied molecular orbital (LUMO) of the dye is then injected into the conduction band (CB) of the meatal oxide. In 1988, Michael Gratzel and co investigators was invented the Ru(II) based dye as promising sensitizer in photoelectrochemical cells.[11] The conversion of visible light to electrical energy depends on nature's principle of photosynthesis. DSSC consists of photo electrode made up of conducting glass plate and coated with porous nano-

[1] *Prof. Ramkrishna More Arts, Commerce and Science College Akurdi, Pune, India*

[2] *Advanced Physics Laboratory Department of Physics, Savitribai Phule Pune University, Pune, India*

* *Email:* kmgadave@gmail.com (K. M. Gadave)

crystalline wide bandgap semiconductor on which dye is adsorbed. A counter electrode is platinum or graphite coated on glass. Electrolyte solution is placed between two electrodes which contains redox couple. The dye on semiconductor absorbs light and eject electron into CB of semiconductor and this electron flows through external circuit thus, electromotive force (EMF) is generated in a cell. The electrolyte receive electron from cathode while oxidized dye receive electron from the electrolyte and regenerated.[12,13] The function of dye is similar to chlorophyll in plants. Recently, TiO_2 nanoparticles (TNPs) are usually used as photo anode materials for the fabrication of DSSCs because of their interesting properties like wide band gap, high surface area for dye adsorption and high stability as photo anode. However, its low electron mobility and transport properties are responsible to find out alternatives to TiO_2.[14]

ZnO is one of the promising alternatives to TiO_2 due to its low cost, easy availability, and simplicity in preparation using simple chemical methods. Its energy-band structure is similar to that of TiO_2. It possesses a higher electron mobility than TiO_2.[15-16] ZnO is II-VI compound semiconductor material having a direct bandgap and a large excitation energy of ~ 3.37 eV and~ 60 meV, respectively at room temperature.[17-20] C. For example, Wang *et al.*[21] improved the dye-sensitized solar cells (DSSCs) with a ZnO nano-flower photo-anode. ZnO is an II-VI semiconductor and the bandgap is 3.37 eV. ZnO-nano-flower film is fabricated by the hydrothermal deposition and it increases the dye loading and harvesting. Good electron conductivity rod of photo-anode efficiency is 1.9%. Zinc chloride aqueous solution was used to obtain seed layer of ZnO on fluorine doped tin oxide (FTO) plate. Aqueous solution of zinc acetate di-hydrate was used to obtain nano-rods and nano-flowers on the seed layer. I-V characteristic of cell was studied using N_{719} dye. For the cell having flower like ZnO the fill factor was 53% and efficiency was 1.9%. While for rod like ZnO fill factor was 36% and efficiency was found 1.0%. The ZnO nano-flower is more efficient than the nanorod.

Among various parameters like photoanode morphology, film thickness, and dye absorption time, dye absorption time is one of the most important parameters influencing the performance of the fabricated DSSCs. The porous nature of the photoanode is responsible for the absorption of dye molecules within the ZnO surface area. As the porosity of film increases, there is an increase in the dye absorption rate hence light absorption gets enhanced. The result in the photoconversion efficiency of the device will be improved.[22] Numerous research reports have reported the effect of dye adsorption time on the performance of the DSSCs. For example, Khadtare *et al.* reported ZnO/TiO_2 photoanode with Rose Bengal dye and N_{719} dye as sensitizer,[23-26] dye adsorption mechanisms in TiO_2 films, and their effects on the photodynamic and photovoltaic properties in DSSCs.[27-28]

In this research work, DSSC photoanode was deposited using commercially ZnO nanoparticles. A seed layer of ZnO deposited on FTO by a simple low-cost doctor blade technique.

The prepared ZnO photoanode was analyzed by using X-ray diffraction (XRD), UV-Visible absorption spectroscopy, and scanning electron microscope (SEM). The 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid (2-HNDBA) photosensitizer was synthesis by a simple chemical method. The prepared ZnO photoanodes were sensitized by the freshly prepared low-cost 2-HNDBA dye. In this work, the novel 2-HNDBA dye was used for the fabrication of DSSC. This dye was not reported till the date as a sensitizer in ZnO based DSSC. The effect of dye absorption time on the performance of ZnO based DSSC was systematically studied.

2. Experimental
2.1. Materials and Methods
The following materials and methods are used for the fabrication of DSSCs,

Materials: Zinc acetate (Sigma Aldrich), sodium hydroxide (Thomas Baker), ZnO Nanopowder (Sigma Aldrich), ethylcellulose (SDFCL), α-terpineol (HPCL), acetylacetone (SRL), p-aminobenzoic acid, HCl, etc.

Methods: (a) CBD Method for deposition of a compact layer. **(b)** Doctor blade method for deposition of the seed layer.

2.2. Synthesis of ZnO barrier layer
The barrier layer of ZnO was synthesized using 0.01 M zinc acetate in 100 mL ethanol. 0.05 M sodium hydroxide was added dropwise in the above solution until just white turbidity appeared. The cleaned FTO plate was dipped into this solution and dried. This process was repeated 10 times, and then the film was dried on a hot plate at 250 °C for 30 minutes.

2.3. Preparation of ZnO seed layer
The thick layer of zinc oxide was prepared on the barrier layer by doctor blade technique.[29-30] 10 g of zinc oxide powders were mixed with methylcellulose (30 wt % metal oxide) and ground for 1 h in mortar and pestle. This mixture was transferred into a screw cap test tube and ethanol was added (approx 25 mL) to form a paste. This paste was sonicated for 4 cycles (each cycle was of 15 minutes.). After the formation of uniform suspension, 1.5 mL acetylacetone and 3.5 mL α-terpineol were added to it, and the paste was stirred with a glass rod and then again sonicated for 2 cycles of 15 minutes. Then FTO was covered with the scotch tape (double layer) and the central 1 cm portion was kept open. Then the drop of paste was placed on the central portion of the plate (at one end) and the uniform layer was obtained by spreading it with another glass plate. The plate was dried at 60 °C in a hot air oven and then another layer was applied similarly. The plate was dried at 60 °C again for 30 minutes, then the scotch tape was removed and the plate was annealed at 450 °C for 1 h where the heating rate of the furnace was adjusted 10 °C per minute.
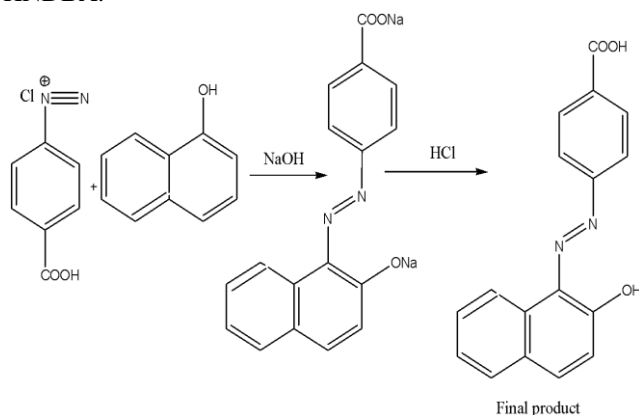
2.4. Synthesis of dyes
The 2-HNDBA dye was prepared as reported by Mann *et al.*[31] The first step of preparation of dye is the diazotization of p-aminobenzoic acid. When HCl was added to $NaNO_2$ solution

then HNO₂ was formed. HNO₂ reacted with p-aminobenzoic acid to form diazonium salt (Fig. 1.).
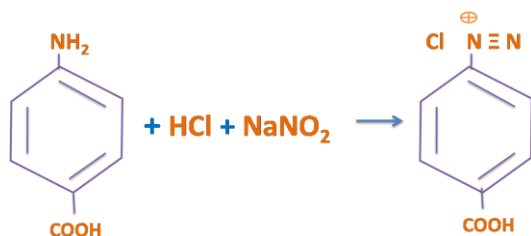
Fig. 1 Reaction of diazonium salt of p-aminobenzoic acid to form 2-HNDBA.

Fig. 2 Reaction for β-naphthol 1-Azo p-ammino benzoic Acid Dye.

The diazonium salt of p-aminobenzoic acid then reacted with the alkaline solution of beta naphthol (1:1 molar proportion) to form the red-colored sodium salt of azo dye. The salt on acidification gave insoluble orange-colored dyestuff, which was recrystallized from ethanol and used in the present study (Fig. 2). The percent practical yield of the dye was 70.76% and the melting point of this dye was 187 °C. Purity of dye was checked by thin layer chromatography (TLC) method. TLC showed a single spot on plate having R$_f$ (retardation factor) value 0.71. It matches with the reported value.[31] Therefore, we concluded that the final product obtained is the expected dye molecule i.e. 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid dye. The IUPAC name of synthesized azo dye is 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid. The dye is synthesized concerning its structure. It is reported that when the dye molecule is planar, consists of a binding group, and has conjugated double bonds, then it gives a good efficiency in DSSC.[32-34] In the synthesized molecule, –COOH and –OH can act as a binding group to oxide while two benzene rigs are in conjugation with each other through the azo bond. 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid dye is abbreviated as 2-HNDBA.

The advantages of 2-HNDBA dye are the following. It is cheap and simple in the process. This means that the dye is cost-efficient. It is an environment friendly in nature. It is easily soluble in alcohol like methanol, ethanol, etc. It shows a maximum absorption at 488 nm (Visible Region). The above advantages make the 2-HNDBA dye as an efficient sensitizer for DSSCs.

2.5. Sensitization of dye
The 2-HNDBA dye solutions were prepared using 50 mg of prepared dye in 25 mL methanol. It was completely dissolved to form a clear solution. ZnO coated films were immersed in the dye solution for 60, 120, 180, and 240 minutes and taken out. Then films were dried in a dryer at 60 °C for 10 minutes.

2.6. Preparation of Counter Electrode
The counter electrode was prepared using graphite powders, which were prepared by applying a thin coat of graphite on the FTO conducting plate (HB pencil lids were used as a source of graphite). The uniformity was checked by checking the conductivity of the film, which was around 3-5 Ohm only. These electrodes with carbonaceous materials have various advantages such as high electron conductivity, decreased corrosion resistance, and minimum cost required than the platinum electrode.

2.7. Preparation of electrolyte solution
Polyiodide was used as an electrolyte in the present study. It is an aquesou solution. 0.5 M potassium iodide and 0.05 M iodine were prepared in 100 mL distilled water. The solution was stirred well and pH was maintained to 7.

2.8. Construction of dye-sensitized solar cell
The DSSSC devices were fabricated using ZnO based photoanode, 2-HNDBA dye, poly-iodide as an electrolyte, and graphite coated FTO substrate as the counter electrode. Two drops of electrolyte were placed on ZnO film in the spacer cavity and the cathode was placed on it. The cathode and anodes were held tightly with the help of clamps. The electrical connections were established from cathode and anode with the help of alligator's clips and wires. This prepared device was characterized by photovoltaic measurement.

3. Characterizations
The deposited ZnO photoanodes were characterized by XRD, UV-Visible absorption spectroscopy, SEM, energy dispersive X-ray spectroscopy (EDS), etc. for the confirmation of structural optical and morphological properties. The structural properties of the ZnO photoanode were performed to investigate the crystal structure using an X-ray diffractometer (model Bruker D8 with CuKα radiation of wavelength 1.54 A°).

The optical properties of 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid photosensitizer sensitized ZnO films were studied by JASCO V-670 UV-Vis spectrophotometer. The scanning electron microscopy was used to investigate the

Table 1. Unit cell parameters of the ZnO photoanode.

2Ө	d (nm)	Peak intensity	[hkl]	Unit cell parameters	
31.86	2.807	65.39	[100]		Observed
34.49	2.599	47.36	[002]	A	3.2411 Å
36.34	2.471	100.0	[101]	C	5.1963 Å
47.62	1.908	17.82	[102]	Unit cell volume	4.272×10^{-23} cm^3
56.68	1.623	34.00	[110]	Dx	5.752 g/cm^3
62.9	1.477	25.02	[103]		
66.45	1.406	5.42	[200]	Average crystal size	35. 87 nm
68.02	1.378	23.09	[112]		
69.16	1.358	11.94	[201]		
72.61	1.301	2.54	[004]		
77.03	1.237	3.50	[202]		

surface morphology of the 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid synthesized ZnO photoanodes by using JSM-7600F. The photovoltaic performance of 4-[(2-hydroxy-1-naphthyl) diazenyl] benzoic acid sensitized ZnO films was studied under the illuminated conditions by using a Keithley 2400 source meter and solar simulator (ENLITECH Model SS-F5-3A) with an incident light intensity 100 mW/cm^2.

## 4. Results and Discussion
4.1. Purity of ZnO
The purity of zinc oxide is calculated using chemical methods, where Zn(II) content was analyzed by complex metric titration. It is agreed with the molecular formula of zinc oxide.

4.2. X-ray diffraction Analysis
Fig. 3 shows the XRD pattern of ZnO. All peaks match with the Standard JCPDS card no. 36-1451, having 2Ө equal to = 31.86, 34.49, 36.34, 47.62, 56.68, 62.9, 66.45, 68.02, 69.16, 72.61 and 77.03 corresponding to (100, (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202)). This also confirms the hexagonal crystal structure (Patil et. al, 2010).[35-39] The extra peaks are not observed, which confirm that ZnO is pure having a single crystalline phase.[40] The average particle size of powder ZnO was 35.87 nm estimated by using the Debye-Scherer formula.

The structural parameter a and c were calculated from the interplanar distance lattice constants and it was 3.241 and 5.196 Å. The unit cell volume was calculated from the lattice constants a and c and found to be 4.272×10^{-23} cm^3. For the

determination of ZnO by using the unit cell volume, X-ray density (Dx), and it was found to be 5.752 g/cm^3. These values are in good agreement with previously reported values of Zinc Oxide have hexagonal unit cell structure.[5,6, 41]
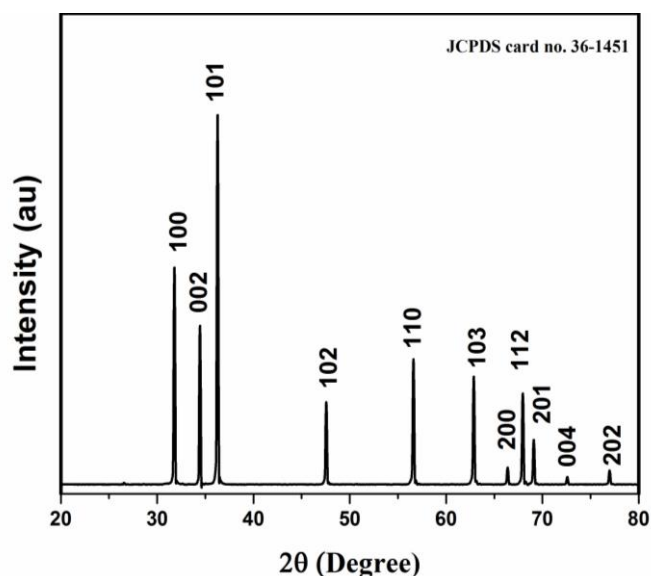
**Fig. 3** XRD patterns of the ZnO photoanode.

4.3. Optical Properties
The optical properties of the ZnO photoanode were measured by using JASCO UV-Vis. Spectrometer within the range of 200 to 800 nm. Fig. 4. shows the optical absorption of the 2-HNDBA dye. UV-Visible absorbance spectra of synthesized

Table. 2 Photovoltaic parameters of 2-HNDBA dye-sensitized ZnO photoanode based DSSCs for various dye absorption time.

	Photovoltaic parameters			
Dye Adsorption Time (minutes)	60	120	180	240
V_{OC} (V)	0.332	0.347	0.346	0.350
I_{SC} (mA/cm²)	1.927	2.896	2.488	2.442
FF	0.44	0.53	0.51	0.51
Efficiency (%)	1.09	2.08	1.71	1.69

dye were recorded where dye was dissolved in methanol at the appropriate concentration. The absorbance spectra of the dye in methanol show a maximum absorbance at 488 nm.
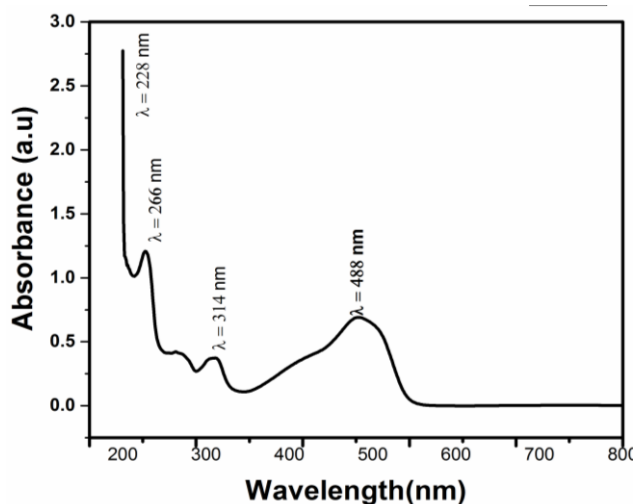

Fig. 4 Absorbance spectra of 2-HNDBA dye.
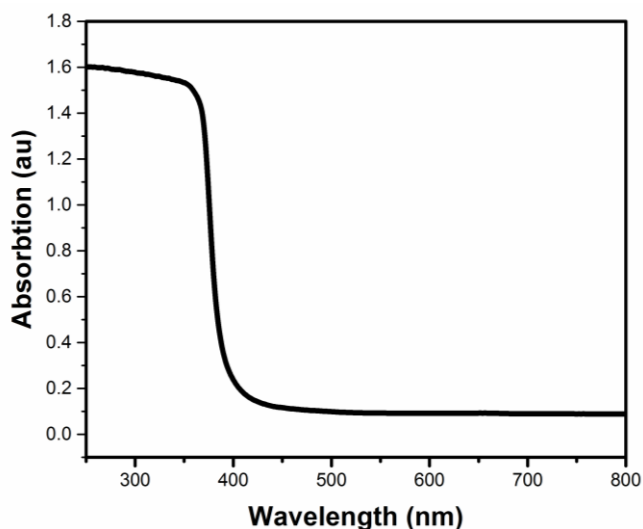
Fig. 5 Absorbance spectra of the ZnO photoanode.

Fig. 5 shows the optical absorption of the ZnO photoanode. The bandgap of ZnO photoanode was obtained from diffuse reflectance spectra in absorbance mode and recorded in the solid-state. Fig. 6 shows the tauc plot of the ZnO thin film. The band gaps of ZnO were found to 3.24 eV. The absorption of energy at the bandgap corresponds to the electronic excitation of an electron from a valence band to the gaps of metal oxide

that are in good correlation with the bandgap reported for this oxide.[42, 43]
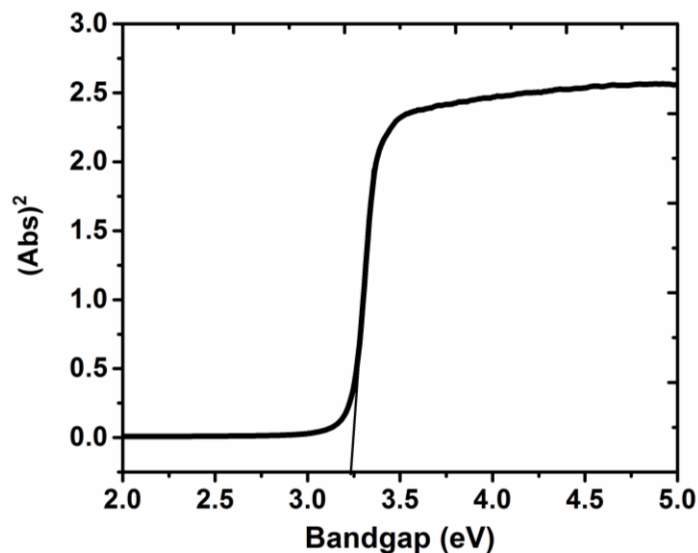
**Fig. 6** Tauc plot of the ZnO photoanode.

The mass loading (or dye uptake mol/gm) of the 2-HNDBA dye is determined from the absorption spectrum of the dye at various dye adsorption periods. In pure methanol, the dye from the ZnO film was desorbed and the spectrum was recorded. In Fig. 7., these are presented. The loading of the amount of dye on the ZnO film was determined from the dye absorption. For 60, 120, 180 and 240 minutes of dye loading time the amount of dye loaded was respectively 0.185, 0.223, 0.244, 0.256 mg/cm².

4.4. Scanning electron microscopy (SEM) aalysis

Fig. 8 (a) shows the SEM image of the ZnO barrier layer. We have obtained the barrier layer by chemical deposition method till film is slightly visible on the FTO plate. The thickness of ZnO barrier layer was approximetly 1.5-2.0 micron. It shows the irregular shape rod-like morphology. The growth orientation of the ZnO nanorod was perpendicular to the substrate. The particles are not uniform in length and diameter. Fig. 8 (b) shows the SEM images of the ZnO photoanode prepared by the doctor blade method. The surface morphology played a significant role in dye adsorption.[44] Fig. 5 shows the porous nature of the ZnO photoanode. It improves the dye absorption rate and thus the photoconversion efficiency of DSSC was enhanced.[44]
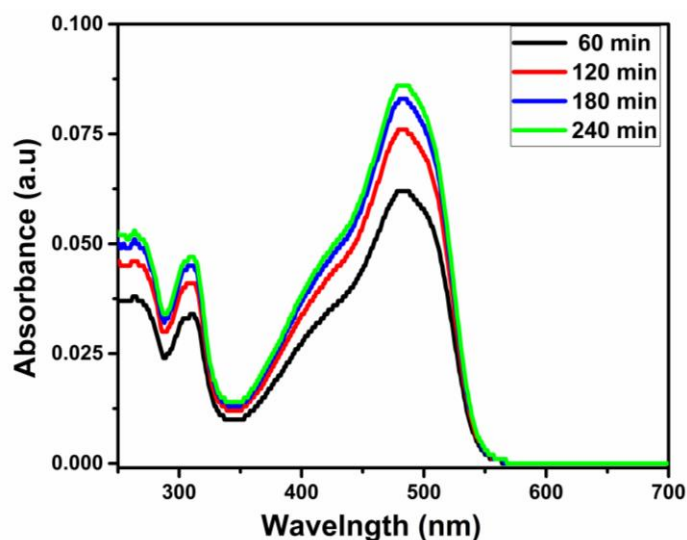
Fig. 7 Absorbance spectra of 2-HNDBA dye for 60, 120, 180 and 240 minutes respectively
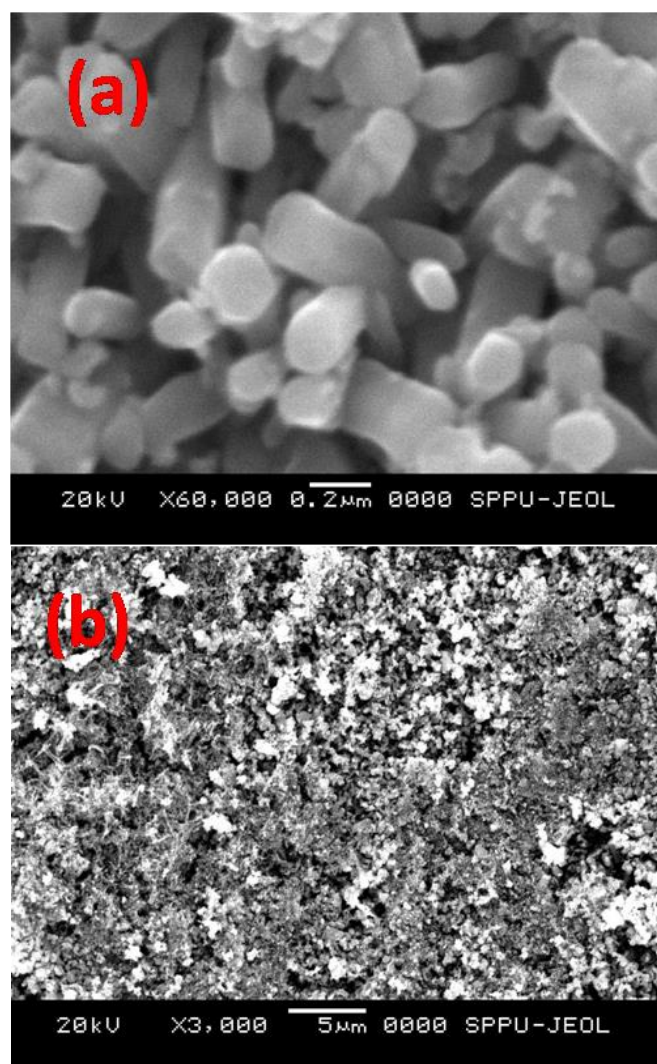
Fig. 8 SEM image of (a) the barrier layer of ZnO film and (b) ZnO photoanode (Top view).

4.5. Photovoltaic measurements

Fig. 9 show I-V characteristics DSSCs based on ZnO photoanode sensitized with 2-HNDBA dye. ZnO photoanode was sensitized for various time, the adsorption rates (60, 120, 180, and 240 min) were tested for photovoltaic performance under one sun condition. Similar results on the effect of dye adsorption time on ZnO/TiO_2 photoanode with Rose Bengal dye and N_{719} dye as sensitizer were obtained by Khadtare *et al.*[26] The photovoltaic parameters such as V_{OC}, short-circuit J_{SC}, fill factor (FF) and photoconversion efficiency (η) of DSSCs are discussed in the following paragraph and also summarized in Table 2.
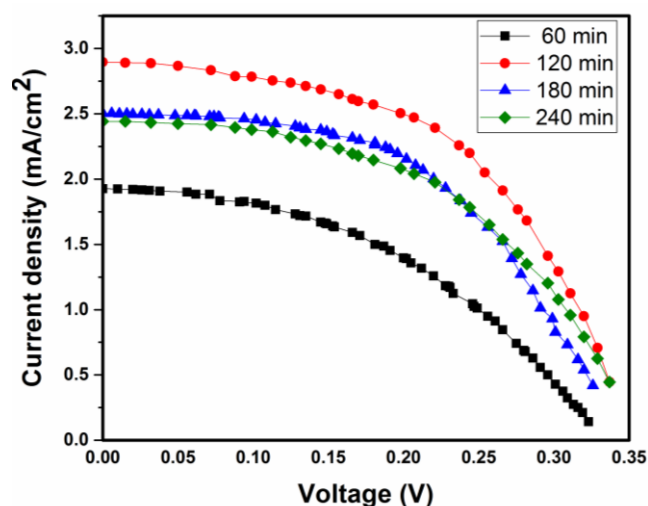
Fig. 9 I-V curve of ZnO film at different dye adsorption time of 2-HNDBA dye

The ZnO photoanode sensitized for 60 min dye absorption time shows a poorer performance than other cells. Since 60 minutes dye absorption time is less for the adsorption of dye on ZnO and in this time all adsorption sites of ZnO might not be occupied by the dye molecules.[45] Thus, such cells displayed a lower performance. The photo-conversion efficiency was improved with increasing the dye adsorption time from 60 to 120 minutes. From it is concluded that the adsorption time of 120 minutes is sufficient for occupying all surface adsorption sites on ZnO. The adsorption time less than 120 minutes might not be sufficient to occupy all surface adsorption sites of ZnO by the dye. Thus, cell with such a photo-anode where dye adsorption time is 120 minutes showed the best performance. Further increasing the dye absorption time to 180- 240 minute, the photoconversion efficiency of the device was reduced slightly. Because multilayer adsorption of the dye created the barrier for the transfer of an electron from dye molecule to ZnO (Fig. 10.), such barrier reduced the photovoltaic performance of the cell.
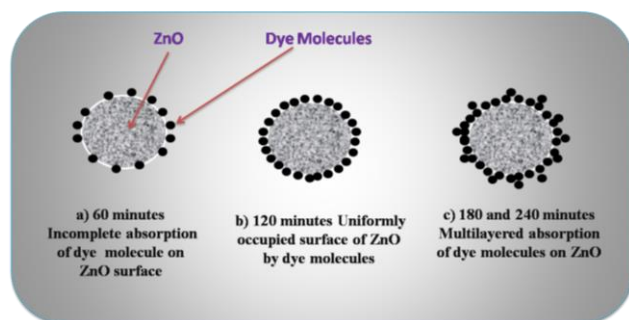
Fig. 10 Effect of dye adsorption time on ZnO surface.

Thus, the above study confirmed that the cell with 2-HNDBA dye-sensitized for 120 min dye absorption time shows a high performance as compared to other cells, which show 0.347 V Voc, fill factor 53 % and overall conversion efficiency of 2.08 %. The current study concludes that dye absorption time played an important role on the performance of DSSCs.

5. Conclusion
In summary, dye-sensitized solar cells (DSSCs) were successfully fabricated using the low-cost 2-HNDBA dye. ZnO barrier layer was successfully grown by chemical bath deposition (CBD) method. The doctor blade method was employed to prepare the ZnO seed layer. The XRD pattern confirmed the hexagonal wurtzite structure of ZnO. The 2-HNDBA dye was successfully loaded on a porous ZnO photoanode at various dye absorption time and studied its photovoltaic properties. 2-HNDBA dye consists of polar functional groups, i.e., –COOH and –OH in their structure and planar. Thus, the above study confirmed that the cell with 2-HNDBA dye-sensitized for 120 min dye absorption time shows a high performance as compared to other dye absorption time rates, which shows a Voc of 0.347 V, fill factor 53 % and the overall conversion efficiency of 2.08 %. The current study concludes that the dye absorption time played an important role in the performance of 2-HNDBA dye-sensitized DSSCs.

Acknowledgment

Support information
Not applicable

Conflict of Interest
There are no conflicts to declare.

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Author information

Shital S. Rakhunde *received her master's degree in Analytical Chemistry and her M. Phil. degree is in chemistry from the Savitribai Phule Pune University, Pune. Title of her thesis at M. Phil. degree is ZnO photoanode based dye sensitized solar cell with Azo Dyes of P-amino benzoic acid with β-Napthol and 8-Hydroxyquinoline.*

Kisan Mahadeo Gadave *Received his master's degree in Inorganic Chemistry and his Ph. D. in chemistry from the Shivaji University Kolhapur, India. He joined as assistant professor to Pune Districts Education Association's B. G. College Sangvi, Pune, India and presently working at Prof. Ramkrishna More College Akurdi. Presently he is Associate professor in the same institute. He received M. Phil. guide recognition in chemistry from Savitribai Phule Pune University, Pune. Presently he is working on deposition of thin films and their applications in PEC cell and in DSSC.*

Dnyaneshwar Rambhau Shinde *Received his master's degree in Inorganic Chemistry and his Ph. D. in Environmental Science from the Savitribai Phule Pune University, Pune, India. He joined as assistant professor to Prof. Ramkrishna More College Akurdi, in 1999 and now he is Associate professor in the same institute. He received Ph. D. guide recognition in chemistry from host university. Presently he is working on synthesis of nanomaterials and its applications in DSSC, photo catalysis and in gas sensing.*

Pankaj K. Bhujbal *is a research fellow in Physics at the Advanced Physics Laboratory, Department of Physics, Savitribai Phule Pune University, Pune, India with Dr. Habib M. Pathan. He received his master's degree in Physics from the Fergusson college, Pune. His research interests include Thin films, Quantum Cascade Laser and DSSCs.*

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