Fabrication of DSSC with Nanoporous TiO$_2$ Film and Kenaf Hibiscus Dye as Sensitizer

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Abstract- Here, we have demonstrated the fabrication of a DSSC with much higher efficiency in comparison to others in its category using nanoporous Titanium Dioxide (TiO$_2$) film as active material. The dye which acted as photo sensitizer, was extracted from Kenaf Hibiscus (Hibiscus Cannabinus) flower by the indirect hydronic heating method. To the best of our knowledge, dye extracted from kenaf hibiscus has not been used as photosensitizer for DSSC application till date. The TiO$_2$ film was fabricated on Indium Tin Oxide (ITO) coated glass and was annealed for 20 mins at 400°C to remove the lattice defects by recrystallization. Further it was absorbed in dye and the dye absorbed TiO$_2$ film acted as photo anode. Another ITO coated glass was coated with platinum solution to minimize the resistive loss and better collection of electrons. This served as counter electrode (cathode). The Atomic Force Microscopy (AFM) was used to analyze the morphology of the device which confirmed the presence of TiO$_2$ nanoparticles (NPs). High Resolution X-Ray Diffraction (HRXRD) study revealed the crystalline structure of TiO$_2$ film. The optical absorption study confirmed the working of the solar cell in ultraviolet-visible (UV-Vis) region. The light intensity vs voltage and current, current vs voltage studies were used to calculate different parameters, efficiency and overall performance of the cell. An optimal blend of acetonitrile (solvent) (50-100%), 1,3-dimethylimidazolium iodide (10-25%), iodine (2.5-10%) and lithium iodide, pyridine derivative and thiocyanate was used as electrolytes in the DSSC. The DSSC showed a maximum conversion efficiency of 2.87%.

Keywords: DSSC, TiO$_2$, Kenaf Hibiscus, electrolyte, nanoporous.

1. Introduction

Fossil fuels are getting exhausted day by day very rapidly. Due to this, the alternative energy sources are gaining much popularity. Solar energy serves the huge contribution in the field of renewable and alternative energy resources. Presently, extensive research works are going on worldwide to make efficient solar cells, including inorganic and organic types. The organic material based solar cells find much popularity due to their low cost, flexibility and eco-friendliness. Depending on the materials, structures and fabrication techniques, three generations of solar cells have been evaluated till date[1]. Dye sensitized solar cells (DSCC) are classified as third generation solar cells, that converts the light energy to electricity, based on the sensitization of wide band gap semi-conductors[2]. They are gaining much interest due to their low cost, flexibility, biodegradability, short production time and mostly they can produce electricity even in low light. But the main problem with DSSC is that it shows very less efficiency [3, 4]. Ever since, O'Regan and Grätzel invented the first DSSC in 1991 in the Laboratory of Photonics and Interfaces in Ecole Polytechnique Federale de Lausanne, Switzerland by the successful combination of nanostructured electrodes and efficient charge injection dyes[5]. The performance of the cell mainly depends on the photosensitizer. The absorption spectrum of the dye and the anchorage of the dye to the surface of TiO$_2$ play an important role for determining the efficiency of the cell[6]. The DSSC primarily comprised of photoelectrode, redox electrolyte and counter electrode[7]. Other materials include transparent conducting oxide and sealing agents. DSSC components have gone under
various developments over the years in order to enhance the efficiency of the cell [8,9,10,11,12].

In this paper, DSSCs were prepared using natural dye extracted from kenaf hibiscus flower which showed an efficiency of 2.87%. Others reported maximum conversion efficiency of DSSC using hibiscus were ranging from .07% to 1.1.4%. [7, 13, 14, 15] and using other natural dyes the efficiencies were ranging from .16 % to 4%. [4, 15, 16, 17, 18, 19].

2. Material and Methods

Fabrication of Dye Sensitized Solar cell involves the following steps. Two slides of ITO coated glass (2cm × 2 cm) were cleaned with Ethanol (C₂H₅OH) and de-ionized (DI) water(Fig.2(a)). When the slides were dried due to natural evaporation, the conducting side of the glass was observed to have a resistance of 20-25 Ω on average. A fine blend of nanoporous TiO₂ powder (Global Nanotech) and acetic acid (CH₃COOH) (Nice chemicals) (1:1) was prepared. A small portion of the conductive side of an ITO coated glass piece was covered with scotch tape to avoid generation of film of TiO₂ during the fabrication process(Fig.2(b)). The blend was coated on the conductive side by using tape casting method which formed a film of TiO₂[7, 20, 21]. The effective area on the conductive side of the ITO coated glass slide was about 1.5cm × 1 cm for the film fabrication. After the evaporation of CH₃COOH naturally, a film of nanoporous TiO₂ was grown. The scotch tape was removed when the film was dried. Then the sample was annealed on hot plate for 20 mins at 400°C(Fig.2(c)).Fig.2(d) represents the annealed sample. Meanwhile, Dye was extracted from kenaf hibiscus flower petals (Fig. 3(a)) using pure ethanol (99.9%) by the indirect hydronic heating method(Fig. 3(b)-(d))[22]. The annealed TiO₂ film coated sample was dipped into the dye solution for 10 min (Fig. 4(a)). The TiO₂ film turned to a deep blue colour after soaking the dye. Afterwards, the dye soaked TiO₂ electrode has been taken out of the dye solution and washed with de-ionized (DI) water (Fig. 4(b)). To make the counter electrode, another ITO coated glass slide was coated with platinum solution (Platisol, Solaronix) on the conducting side.

Once both the samples dry off completely they were clamped together facing two conductive sides such that a conductive portion in both the slides was available to connect the measurement probes (Fig.4 (d)).The cell was filled with liquid electrolyte (HI-30, solaronix) by injecting it between the TiO₂: dye electrode and the counter electrode (Fig.4 (c)). The optimal electrolyte solution contains 50-100% acetonitrile (solvent), 10-25% 1,3-dimethylimidazolium iodide, 2.5-10% iodine and it also consists of lithium iodide, pyridine derivative and thiocyanate. Thus the device was ready to analyze different electrical characteristics. The schematic diagram of the cell is represented by Fig. 1. Fig.5 depicts the Working mechanism of the DSSC.
Figure 3. (a) Kenaf Hibiscus flowers, (b) flower petals dipped in ethyl alcohol, (c) extraction of dye by indirect hydronic heating method (d) filtration of dye from the residues.

Figure 4. (a) TiO₂ film sample dipped in dye, (b) cleaning with DI water, (c) injection of electrolyte, (d) Connecting the measuring probes.

Figure 5. Working mechanism of the DSSC

3. Results and Discussion

3.1. Structural Characterizations

The surface morphology of the dye absorbed nanoporous TiO₂ film was analyzed using Atomic Force Microscopy (AFM). Fig. 6(a) and (b) shows the 2D and 3D AFM images of the fabricated dye absorbed nanoporous TiO₂ film. An average of 10 nm TiO₂ nanoparticles was observed in the images. The dye particles were dispersed in the TiO₂ layer. The High resolution X-Ray Diffraction (HRXRD) (Panalytical, X’Pert Pro X-ray diffractometer) pattern of the nanoporous TiO₂ film (Fig. 7) depicts the crystalline structure of the TiO₂ film. All the diffraction peaks refer to anatase and rutile phases (JCPDS No. 84-1285 for anatase and 87-0920 for rutile) [15, 23]. The pattern shows an average size for both the phases.
3.2. Optical Characterizations

The UV-Vis optical absorption measurement (250-1000 nm) was done on the dye, nanoporous TiO$_2$ film and dye absorbed TiO$_2$ film at room temperature by a UV-visible near-infrared spectrophotometer (Lambda 950, Perkin Elmer). The nanoporous TiO$_2$ film shows a peak in UV region (375 nm) (Fig. 8 (a)).

The spectrum of Kenaf Hibiscus dye (Fig. 8 (b)) shows an optical absorption in Uv-vis region having a peak at 378 nm and a small hump at 554 nm. The optical absorption spectra of dye absorbed TiO$_2$ film (Fig. 8 (c)) has two peaks at 385 nm and 548 nm with broad absorption spectra in the visible range which is required for a solar cell. The (αhγ)$^2$ versus ev curves of the nanoporous TiO$_2$ film and dye absorbed TiO$_2$ film are shown by (Fig. 8 (d)). In case of nanoporous TiO$_2$ the main band gap is shown~3.35 eV which was reported by other authors also [24, 25]. But dye absorbed TiO$_2$ film exhibits the band gap at ~ 1.8 eV and ~ 2.24 eV which may correspond to the Kenaf Hibiscus dye. Another major hump is obtained at ~ 3.5 ev which may be due to sub band gap transition of the nanoporous TiO$_2$. The band gap shifting were reported in other journals also [24].
3.3 Electrical characteristics

Fig. 9 (a) and (b) shows the Illuminance vs Voltage (L-V) and Illuminance vs Current (L-I) characteristics of the solar cell respectively at room temperature. The illuminance of incident light was measured by lux meter (HTC LX-101A) and the voltage and current measurement were done by a multimeter (FLUKE 289 TRUE RMS). In both the graphs the values were increasing with the increment of incident light and after a certain limit both voltage and current were saturated. The I-V characteristics study was done using an I-V meter (Agilent technologies) and plotted in graph (Fig 10 (a)). The following parameters are measured as: open circuit voltage (V_{oc}) = 0.4788 V, short circuit current (I_{sc}) = 10.01 mA, maximum power point voltage (V_{mp}) = 0.3670 V and maximum power point current (I_{mp}) = 7.85 mA. Fig. 10 (b) represents the current density vs voltage (J-V) characteristics graph where device active area was 1.5 cm² and short circuit current density (J_{sc}) was 6.6733 mA/cm².

During the calculation of maximum conversion efficiency at room temperature, a solar simulator was used to produce the irradiation (G) of 1000 W/m² i.e. 0.1 W/cm² and air mass (AM) of 1.5.
\[ \text{Fill Factor (FF)} = \frac{V_{mp} \times I_{mp}}{V_{oc} \times I_{sc}} = \frac{0.3670 \times 7.85}{0.4788 \times 10.01} = 0.60 \]

\[ \% \text{Efficiency (}\eta\text{)} = \left( \frac{V_{oc} \times I_{sc} \times \text{FF}}{P_{in}} \right) \times 100\% \]

\[ = \left( \frac{0.4788 \times 10.01 \times 0.60}{0.1 \times 1000} \right) \times 100\% = 2.87\% \]

**Figure 9.** (a) Illuminance vs Voltage (L-V) and (b) Illuminance vs Current (L-I) characteristics of the solar cell at room temperature.

**Figure 10.** (a) Current vs Voltage (I-V) and (b) Current density vs Voltage (J-V) characteristics of the solar cell at room temperature.

- \( V_{mp} = 0.3670 \text{ V} \)
- \( I_{mp} = 7.85 \text{ mA} \)
- \( V_{oc} = 0.4788 \text{ V} \)
- \( I_{sc} = 10.01 \text{ mA} \)

- \( J_{sc} = 6.6733 \text{ mA/cm}^2 \)
- Device Active Area = 1.5 cm\(^2\)
4. Conclusion

In this paper, we have demonstrated the extraction of the dye from the kenaf hibiscus flower by indirect hydronic heating method and also the fabrication of nanoporous TiO$_2$ film based DSSC. An optimal blend of electrolytes was used to maximize the cell efficiency. ITO coated glass was used for both the front and counter electrodes and a platinum solution coating was used on the back electrode to maximize the collection of electrons and to reduce the current loss. The AFM and HRXRD studies confirmed the nanoporous morphology of the cell and the crystalline structure of the TiO$_2$ film respectively. The optical absorption study revealed the absorption properties of both the TiO$_2$ and dye individually and their absorption contributions in the blend. The I-V characteristics graph defined the values $V_{oc}= 0.4788$ V, $I_{sc}= 10.01$ mA, $V_{mp}= 0.3670$ V and $I_{mp}= 7.85$ mA. The maximum conversion efficiency of the cell was measured to be 2.87 % which is much higher than other research works reported earlier. The absorption spectra of solar cell was in UV-Vis region which means the cell operated in UV to visible region of light which has increased the efficiency. The optimum blend of the electrolytes served a very good and fast electron exchanger between the active layers and platinum coating on the counter electrode reduced contact resistance and also increased collection of electrons and their mobilities which increased the overall cell efficiency.

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References