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# A Comparative Study of a Novel Technique for the Fabrication of Dye Sensitized Solar Cells Using Nano$TiO_2$ and Different Dyes

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**ABSTRACT**

Dye-sensitized solar cells (DSSCs) have gained widespread attention in recent years because of their low production cost, ease fabrication and tunable optical properties, such as colour and transparency. Now-a-days natural dye was used to sensitize the electrode and the counter electrode was prepared by the help of carbon black. In this study we report molecularly engineered different dyes (henna, pomegranate and beet root) and nano$TiO_2$ in the DSSCs, which features the prototypical structure of a donor–π-bridge–acceptor and maximizes electrolyte compatibility with improved light-harvesting properties. Bulk$TiO_2$ of sizes 150 micron were converted to nano$TiO_2$ particles having sizes less than 20 nm using planetary ball mill. Our design consists of a lattice of modulated-diameter nano$TiO_2$ particles and interstitial regions filled with electrolyte. This provides not only light trapping and absorption enhancement, but offers improved electrical transport through the nano$TiO_2$ particles. It is observed that when frequency increases both capacitance and resistance decreases. At certain point capacitance it maintains a steady state and resistance is nearly equal to zero. This is due to the internal resistance and the steady state capacitance of the cell. It conforms that the fabricated dye sensitized solar cell works like a conventional cell. It is found that henna and pomegranate dyes shows better energy conversion efficiency than beet root dye.

## INTRODUCTION

The world is now shifting from the conventional energy sources to renewable energy to meet the energy demand. Among the sustainable technologies, photovoltaic technology is regarded as the most efficient [1]. It is based on the concept of charge separation at an interface of two materials of different conduction mechanism [2]. Dye sensitized solar cells (DSSCs) have received considerable attention and a remarkable high conversion energy efficiency of nearly 10% using crystalline mesoporous $TiO_2$ film [3], in which the optical absorption and charge separation takes place. The assembly of a dye-sensitized solar cell is based on a layered structure, which consists of two transparent glass plates with a Transparent Conductive Oxide (TCO) on it, placed parallel to each other and spaced of about 40 μm apart. On one of the plates is applied a nanocrystalline $TiO_2$ layer coated organometallic photosensitive dye – this collection, retrieve in the cell function photo-anode (illuminated anode). The surface of the other glass

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plate with TCO is usually coated with nanoplatinum, which is a catalytic layer – this arrangement is meant for the cell cathode. The space between the plates is filled with an electrolyte containing a redox system $I^-/I_3^-$. Each component shows the dependence between many other materials. If at least one component in DSCCs is changed, e.g., the dye, the composition of the electrolyte, the particle size of the $TiO_2$ or the film thickness, the DSSC cell requires adjustment to ensure finest system management [4]. The electrolyte and the dye are essential components of the cell. The mission of the counter electrode is to gather electrons flowing from the outer current and to catalyse the reduction of the triiodide ions. Platinum is the most common material used as a counter electrode. Although platinum shows a high catalytic activity, its scarcity in resources, high costs and corrosion opportunity through a triiodide solution, inhibit its application on a large scale in the future. For this reason, there is a need for research on alternative materials for platinum, which are characterized by electrochemical activity and chemical stability.

Conversion efficiency of DSCCs also depends upon the nature and selection of dye. The DSSC in which ruthenium based sensitizer is used exhibited maximum efficiency of 12% [5], but it is expensive for large scale application [6]. To replace the ruthenium dye many kinds of natural dye have been investigated and tested [7]. Further, in spite of the low efficiency and life span, natural dyes have always attracted the interest of researchers. Easy availability, compatibility and biodegradability with the environment are the major advantages of natural dye [8]. Natural dye extracts generally contain phytochemicals like quinones, flavonoids; anthraquinones, anthocyanin and coumarines, and they play a vital role in DSSC. Presence of functional group like hydroxyl and carboxyl can act as good metal chelators when adsorbed on $TiO_2$. DSSC fabricated from natural dye is environmental friendly and has low cost in comparison to ruthenium dye [6].

A number of papers on DSSCs have been reported in several journals [9-12]; all these DSSCs have less energy efficiency and less durability. The present study is based on a communication of new methodology; the technique adopted here is very simple and user-friendly. This is a green approach as the process does not pollute the environment and involve no toxic discharge, further dye sensitized solar cells (DSSCs) was fabricated economically for better energy efficiency. The process involves the conversion of bulk$TiO_2$ (150 micron) to nano$TiO_2$ of sizes less than 20 nm with the help of planetary ball mill. Natural dye was extracted from beet root (beta vulgaris) and used to sensitize the electrode. The counter electrode was prepared with the help of carbon black. The tri-iodide electrolyte was consisting of KI and $I_2$ in anhydrous ethylene glycol. When light strikes the surface of this DSSC electron transport processes occur in the following five steps: (a) The dye molecules become excited to a higher electron state as a result of photon absorption, (b) Excited dye molecule gives an electron ($e^-$) into the semiconductor layer of nano$TiO_2$, leaving oxidized dye, (c) Then electrons are wander between nanoparticles of titanium dioxide to the glass with a TCO and the external circuit to the counter electrode, (d) $I^-$ ion leads to reduction of excited state of dye molecules, and this ion is oxidized to the $I_3^-$, (e) Triiodide anion is reduced with use of electron from counter electrode. Then the system returns to an energy balance state and is ready to receive the next photon, and the process began again. It is found that with increase in frequency the resistance of the cell decreases, it nearly equal to zero and tends to zero also. So the fabricated dye sensitized solar cells using different dyes (Henna, Pomegranate and Beet root) are working like a conventional cell. Further a different type of DSSC also fabricated using graphite rod by replacing one Indium tin oxide (ITO glass). A comparative study of voltage current relationship also reported with respect to the effect of dyes.

## MATERIALS AND METHODS

The following materials are required for the fabrication of DSSC as given in **Table 1**.

**Table 1.** List of Materials.

| Sr. no. | Material type | Specification | Quantities |
|---|---|---|---|
| 01 | Indium Tin Oxide (ITO) glass | Size=1"x1'', thickness=1 mm, Resistivity <10 ohm | 02 pieces/cell |
| 02 | $TiO_2$ powder | 150 micron | 1 gm/cell |
| 03 | Dyes (three different dyes) | Hena , Pomegranate, and Beet root | 10 gm/cell each |
| 04 | Consumables | Distilled water, toluene, Potassium Iodide, Iodine, anhydrous ethylene glycol, and ethanol. | As per requirements |
| 05 | Stationeries | clips, candle, dropper, conducting wires, and multi meter | As per requirements |

### Preparation of Nano$TiO_2$

Nano$TiO_2$ was prepared with the help of planetary ball mill (RETSCHPM 100). The container in the ball mill (cup) was washed with distilled water, then cleaned with toluene and kept in the hot air oven at 80°C for 5 minutes to dry it properly, and finally kept it in the room temperature for 10 min. Bulk $TiO_2$ (10 g) powder was taken in the container of the planetary ball mill followed by addition of 15-20 ml of toluene to it for wet grinding. The molar mass of $TiO_2$ is 79.866 gm/mol; according to 10:1 (ball to powder weight) ratio, 10 g of powder was taken in the cup and it was grinded for 15 h. with a speed of 300 rpm followed by 5 min. of rest for every 15 min. of operation.

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**Preparation of Photo Electrode**

The working electrode was prepared by the coating of nano$TiO_2$ paste on the ITO glass. Nano $TiO_2$ powder (2 g) was taken in a 50 ml beaker, followed by addition of 3 ml of distilled water and 2 ml of ethanol respectively to it. The mixture was kept and allowed to vibrate vigorously in an ultrasonnicator having frequency of 20 KHz and power 600 Watt for a period of 1 hour, then it was kept in room temperature (25°C) for 24 hours; conducting side of the ITO glass can be identified with the help of a multi meter. The resulting nano$TiO_2$ paste is shown in **Figure 1a**, and was then used for coating on the conducting side of ITO glass surface with a withdrawing speed of 2 mm/s. The coated glass was kept in the room temperature for 1 hour and then in the hot air oven for 1 hour in 100°C.

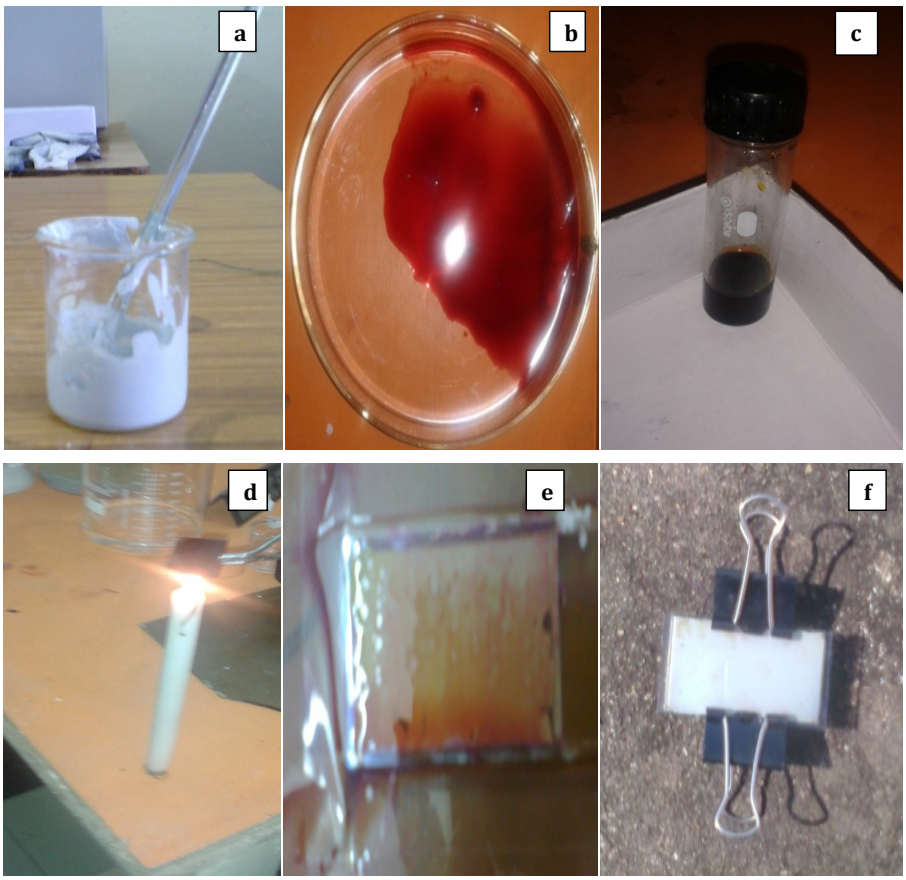


**Figure 1.** a) Nano$TiO_2$ paste b) Beet root dye c) Tri-iodide electrolyte d) Coating of carbon black in ITO glass e) Photo electrode and f) Fabricated DSS Cell.

**Preparation of Dyes, Electrolyte and Counter Electrode**

The Beet root dye (**Figure 1b**) was prepared from the beet root. First beet root was cleaned with water and cut into small pieces. Small piece of beet root (10 g.) were taken and 2-4 ml of distilled water was added to it, then it was grinded for 10 min and filtered to get the Dye. Similar processes were adopted for the preparation of henna dye and pomegranate dye respectively.

The electrolyte was prepared in the room temperature. The tri-iodide electrolyte (**Figure 1c**) was consisting of 0.5 M KI and 0.05 M $I_2$ in anhydrous ethylene glycol.

Counter electrode was prepared by the help of carbon black. The conducting side of another ITO glass was shown to the candle flame, so that carbon black deposited uniformly on it as shown in **Figure 1d**.

**Fabrication of the DSS Cell**

2 to 3 drops of prepared beet root dye was added to the photo electrode, so that dye coated uniformly with nano$TiO_2$. It should be taken care that required quantity of dye should be pour very carefully so that $TiO_2$ should not come out. Then it was kept in the room temperature (25°C) for 1 hour and subsequently in the hot air oven at 100°C for 1 hour to remove moisture. Few drops (2 to 3) of electrolyte was added to it and kept as it is in the room temperature for 2 to 3 hours to dry it completely (**Figure 1e**). DSSC cell was fabricated as shown in **Figure 1f**, by sandwiched of prepared counter electrode with the photo electrode.

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### Instrument and Methodology

The particle size of nano$TiO_2$ was determined using High Resolution Transmission Electron Microscopy (HRTEM model ZEISS EM910) operated at 100 KV, with a 0.4 nm point-to-point resolution side entry goniometer attached to a CCD Mega Vision III image processor.

The conducting side of the ITO glass was found out by the help of multi meter (DT830D). Open circuit voltage and short circuit current also find out by the help of multi meter (Rish Multi 12S).

The resistance, inductance and the capacitance of the cell was measured with variation of frequency by the help of LCR meter (Keysight/Agilent 4284A).

## RESULTS AND DISCUSSION

### Determination of Particle Sizes of nano$TiO_2$

The Selected Area (Electron) diffraction (SAD or SAED) of nano$TiO_2$ is shown in **Figure 2a**. The HRTEM micrograph of nano-$TiO_2$ in different magnification is shown in **Figure 2b-2e**, respectively. It is seen that the particle sizes of nano$TiO_2$ is in the range of 8-16 nm.

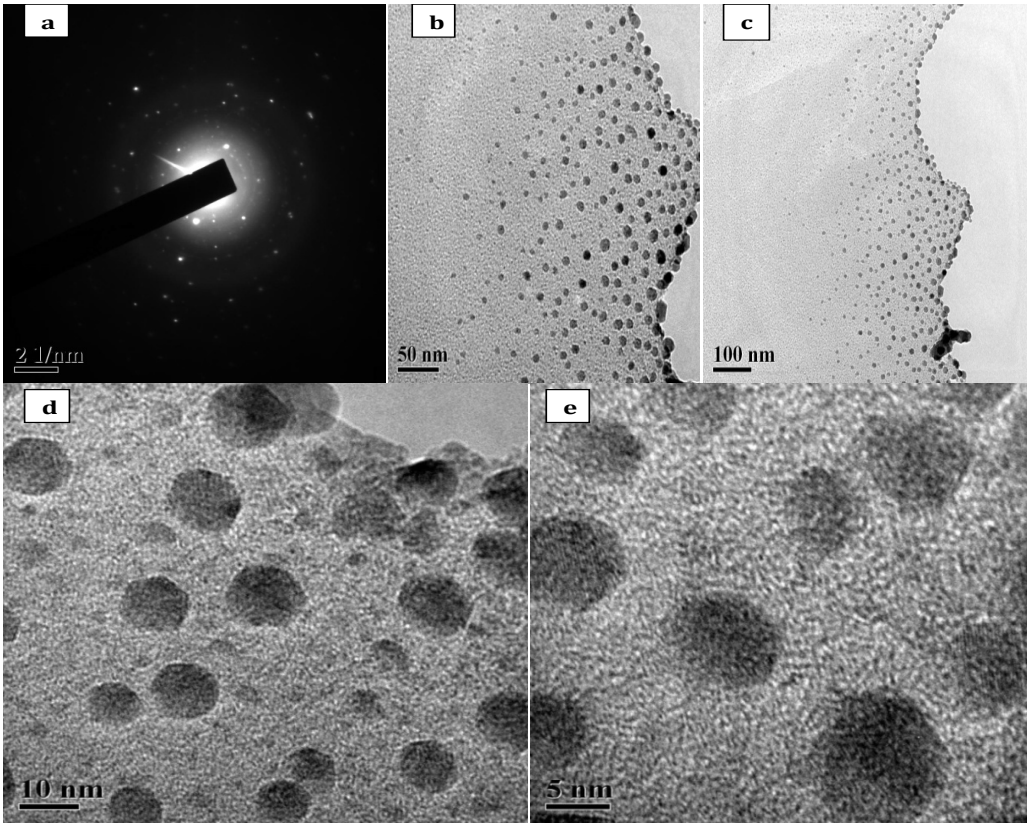


**Figure 2.** (a) SAD pattern of nano$TiO_2$, (b-e) The HRTEM micrograph of nano$TiO_2$ in different magnification.

### Measurement of Photo Voltage

Result from each experiment were presented and analyzed in this section. The readings of Voltage (mV) and Current (mA) which were measured in 298°K and 1 atm. pressure with the illumination of sun light from 10 am to 4 pm is shown in **Table 2**. The reading was taken on 5th December 2017 at KIST campus, Bhubaneswar, Orissa, India, with a geographical position of 20° 7' 43" North and 85° 40' 39" East. Different data of open circuit voltage and short circuit current is reported in **Table 3**, which was measured at room temperature (298°K) in front of fluorescent lamp and incandescent lamp in different conditions. A 40 watt fluorescent lamp and a 100 watt incandescent lamp were taken for measurement respectively.

**Table 2.** Voltage and current Reading under sunlight (beet dye cell).

| Time | 10 am | 11 am | 12 noon | 1 pm | 2 pm | 3 pm | 4 pm |
|---|---|---|---|---|---|---|---|
| Voltage (mV) | 250 | 500 | 550 | 556 | 550 | 545 | 470 |
| Current (mA) | 0.15 | 0.15 | 0.15 | 0.11 | 0.13 | 0.12 | 0.11 |

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**Table 3.** Photo voltage production in front of different Lamps (beet dye cells).

| | | | | | | |
|---|---|---|---|---|---|---|
| **Fluorescent Lamp** | Distance (feet) | 5 | 4 | 3 | 2 | 1 |
| | Voltage (mV) | 26 | 52 | 78 | 95 | 97 |
| | Current (mA) | 0.01 | 0.01 | 0.02 | 0.02 | 0.02 |
| **Incandescent Lamp** | Distance (feet) | 5 | 4 | 3 | 2 | 1 |
| | Voltage (mV) | 11 | 20 | 44 | 59 | 68 |
| | Current (mA) | 0.001 | 0.051 | 0.01 | 0.01 | 0.02 |

It is observed from **Table 3** that with increase in radiation of the sunlight the photo voltage production increases, simultaneous current increases and vice versa. Because of increase in solar radiation, dye can absorb more light and excitation of electron is high so that production of voltage and current becomes high. From **Table 3**, it is observed that with decrease in distance from the lamp the production of photo voltage and current increases, this occurs due to more irradiation of light on the cell.

**Measurement of Different Parameters of Cell**

Different Parameters like Quality factor (Q), Ten delta, Impendence, Parallel C and Parallel R were found with the help of LCR meter (Key sight) at 1 and 30 voltage with variable frequencies which were reported in **Tables 4 and 5** respectively.

**Table 4.** Variation of parameter with respect to frequency (beet dye cell).

| Frequency | Q | Tan delta | Impedance | Parallel C | Parallel R |
|---|---|---|---|---|---|
| 0.10000 | 0.27895 | 3.484 | 321.03 | 0.00133 | 333.28 |
| 0.13895 | 0.29556 | 3.383 | 317.46 | 0.00102 | 331.04 |
| 0.19308 | 0.25498 | 3.921 | 313.11 | 0.00065 | 323.13 |
| 0.26830 | 0.23730 | 4.214 | 301.43 | 0.00045 | 309.80 |
| 0.37218 | 0.22145 | 4.515 | 290.27 | 0.00031 | 297.30 |
| 0.51803 | 0.21879 | 4.570 | 280.90 | 0.00023 | 287.55 |
| 0.71983 | 0.22244 | 4.495 | 274.12 | 0.00017 | 280.82 |
| 1.0002 | 0.23769 | 4.207 | 266.33 | 0.00013 | 273.75 |
| 1.3899 | 0.26754 | 3.737 | 257.98 | 0.00011 | 267.05 |

**Table 5.** Variation of parameters with respect to frequency input volt is 30 V.

| Frequency | Q | Tan delta | Impedance | Parallel C | Parallel R |
|---|---|---|---|---|---|
| 0.1000 | 0.2276 | 4.3922 | 290.66 | 0.0012 | 298.10 |
| 0.1389 | 0.2562 | 3.9019 | 321.13 | 0.0008 | 331.51 |
| 0.1930 | 0.2514 | 3.9771 | 311.14 | 0.0006 | 320.83 |
| 0.2683 | 0.2296 | 4.3540 | 300.87 | 0.0004 | 308.70 |
| 0.3728 | 0.2189 | 4.5679 | 291.11 | 0.0003 | 298.01 |
| 0.5180 | 0.2097 | 4.7671 | 279.20 | 0.0002 | 285.27 |
| 0.7198 | 0.2130 | 4.6933 | 271.71 | 0.0001 | 277.81 |
| 1.0002 | 0.2342 | 4.2690 | 265.08 | 0.0001 | 272.26 |
| 1.3899 | 0.2636 | 3.7936 | 257.39 | 0.0001 | 266.18 |

In **Table 4**, with the increase in frequency from 0.1000 to 1.3899 Hz, impedance, capacitance and parallel resistance vary from 321.03 to 257.98 Ohm, 0.00133 to 0.00011 Faraday (F) and 333.28 to 267.05 Ohm, respectively. In **Figure 3**, the graph was plotted between frequency, parallel C and parallel R, when input is 1 V. Both the graph meets to each other at nearly 150 Hz frequency.

In **Table 5**, with the increase in frequency from 0.1000 to 1.3899 Hz, impedance, capacitance and parallel resistance vary from 290.66 to 257.39 Ohm, 0.0012 to 0.0001 Faraday (F) and 298.10 to 266.18 Ohm, respectively. In **Figure 4**, the graph was plotted between frequency, parallel C and parallel R, when input is 3 V. Both the graph meets to each other at nearly 50 Hz frequency.

It is observed from **Figures 3, 4, Tables 4 and 5** that with increase in voltage the cross-section point of parallel C, parallel R and frequency decreases. It is also observed that with increase in frequency the impedance as well as parallel capacitance decreases. After a certain frequency the capacitance and resistance becomes constant. With high frequency the resistance tends to zero or nearly equal to zero but capacitance becomes zero.

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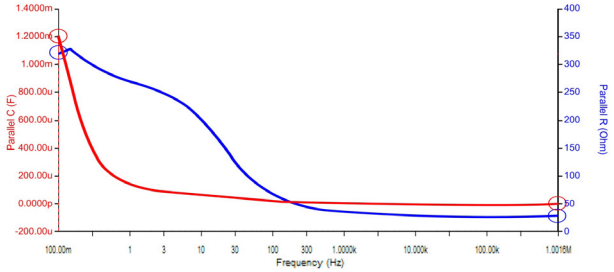


**Figure 3.** Graphs between parallel R and C with different frequency, when input is 1 V.

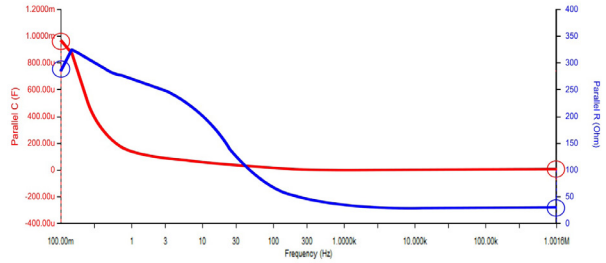


**Figure 4.**Graph between parallel R and C with different frequency, when input is 30 V.

It is also observed from **Figures 3 and 4** respectively that, when frequency increase both capacitance and resistance decreases. At certain point capacitance maintain a steady state and resistance nearly equal to zero, due to internal resistance and steady state capacitance of the cell. It conforms that the fabricated dye sensitized solar cell can work like a conventional cell. **Figures 5-7** represents the graph between current and voltage of three different dyes (henna, pomegranate and beet root) sensitized cells respectively. It is observed in **Figure 5** that, for henna dye the current decreases gradually when the voltage increases then suddenly drops to zero at 500 mV. There is a sharp fall in current when voltage comes to 650 mV. This is due to the sudden resistance build in the cell, the same trend is also observed in **Figures 6 and 7** respectively. **Figure 6** shows the same trend as like **Figure 5**, but in **Figure 7** the current sharply falls at 500 mV. Hence henna and pomegranate dyes have better energy conversion efficiency as compared to beet root dye.

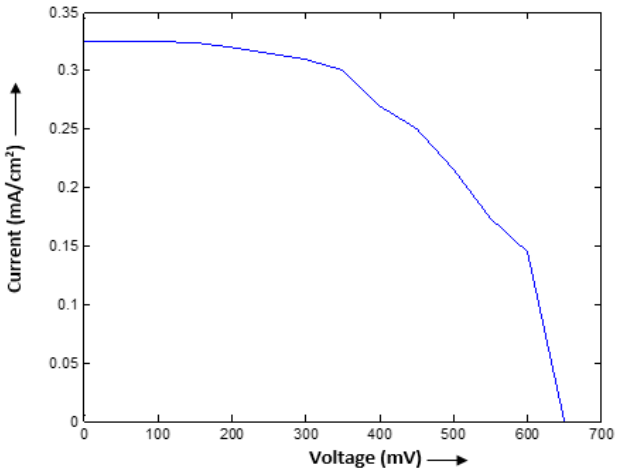


**Figure 5.** Voltage current relationship of DSSC cell using henna dye.

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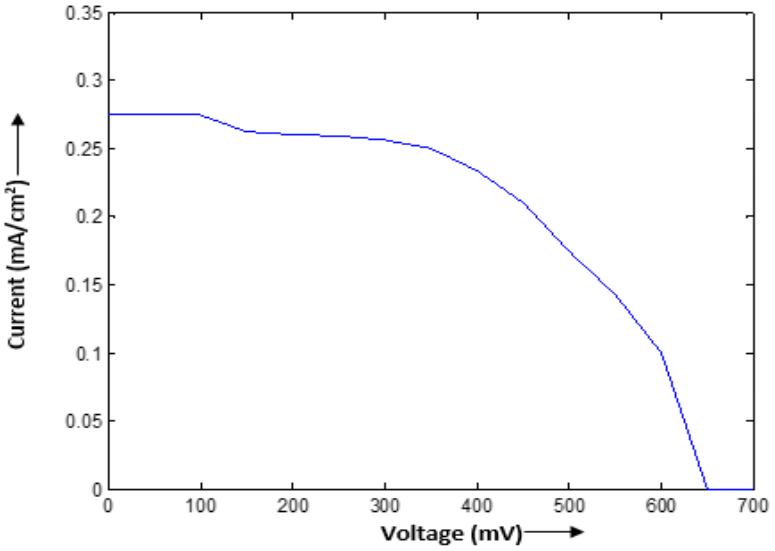


**Figure 6.** Voltage current relationship of DSSC cell using Pomegranate dye.

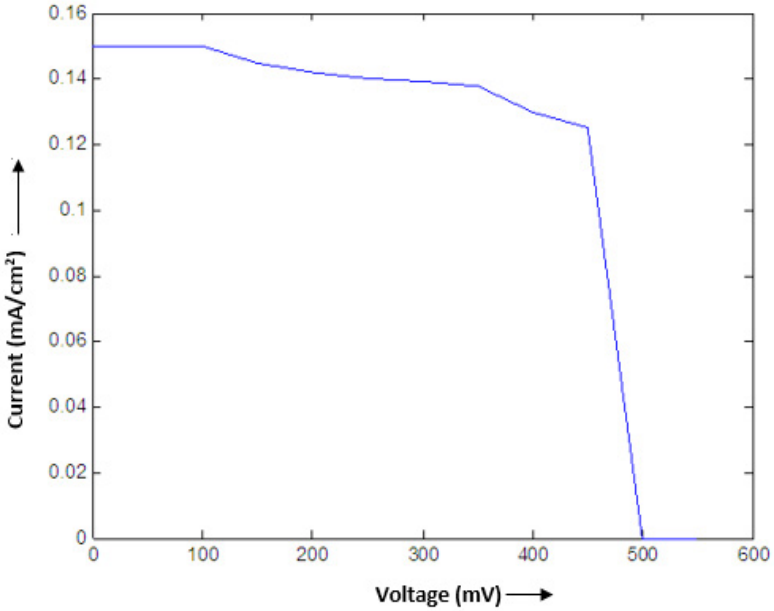


**Figure 7.** Voltage current relationship of DSSC cell using beet root dye.

It is reported that flowers, fruits and leafs are better source of sensitizer than bark and roots [13], so henna and pomegranate dyes are better sensitizer than beetroot dyes. Quinone dye present in henna. Pigment present in beetroot dyes is betalain, which includes betaxanthin and betacyanin. Betacyanin is the major component in beetroot. This betacyanin dye sequentially consists of betanin and indicaxanthin [14]. Low efficiency of beetroot dyes is due to poor interactive nature of betalain with $TiO_2$ [15]. Although, there are reports on pure betanin (dye isolated from core dye betalain that forms the major pigment in beetroot) as a potential sensitizer in DSSC, decay of betanin dye is a major factor which leads to the poor efficiency of DSSC [16]. The rate of decay is influenced by presence of oxygen, pH, light exposure, and temperature. It has also been reported that presence of metal cation ($Ti^{4+}$) will enhance the rate of decay of Betanin [16]. This might be the cause behind low efficiency of beetroot dye. Further beetroot dye is easily coagulated and degraded in the electrolyte and hence sensibly not suitable for cell fabrication. One of the major drawbacks of beetroot is the dye aggregation on $TiO_2$ which may lead to quenching of charge carriers resulting in poor performance of the dye [17]. However, anthocyanin dye present in pomegranate is a relatively small molecule with carboxyl and hydroxyl functional groups. They form stronger bond with $TiO_2$, and it contributes to the power conversion efficiency of the cell. As a matter of fact optical band gap of this dye is low and it will also contribute to the fast regeneration of dye in the presence of $I^-/I_3^-$ redox electrolyte leading to enhancement of fill factor of DSSC. Anthocyanin is the major pigment in pomegranate. It is observed that dyes extracted from pomegranate have least band gap which tends to facilitate fast electron movement to the conduction band of $TiO_2$ [16]. Kavitha et al. [18] have reported that beetroot, henna and pomegranate dyes show a considerable shift in absorption edge towards longer

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wavelength when coated on $TiO_2$. A shift towards longer wavelength would enhance the light harvesting capacity and hence the photo current of the cell. This also indicates the partial chemical bonding of dye with $Ti^{4+}$ of $TiO_2$, leading to the formation of dye-$TiO_2$ complex. Apart from anthocyanin, pomegranate also contains flavylium which strongly binds with $Ti^{4+}$ [15]. These evidences indicates that henna and pomegranate dyes are better sensitizer and have better energy conversion efficiency as compared to beet root dye, which also directly correlated to our experimental findings. It also suggests that partial chemical bonding of this dye with $Ti^{4+}$ of $TiO_2$ possible, leading to the formation of dye-$TiO_2$ complex. Titanium dioxide ($TiO_2$) is taken to be very close to an ideal semiconductor for photo catalysis because of its high stability, low cost and safety toward both humans and the environment. Various investigations have established that $TiO_2$ is much more effective as a photo catalyst in the form of nanoparticles than in bulk powder [19]. When the diameter of the crystallites of a semiconductor particle falls below a critical radius of about 10 nm, each charge carrier appears to behave quantum mechanically [20] as a simple particle in a box. As a result of this confinement, the band gap increases and the band edges shift to yield larger redox potentials [21]. However, the solvent reorganization free energy for charge transfer to a substrate remains unchanged. Because of the increased driving force and the unchanged solvent reorganization free energy, the rate constant of charge transfer in the normal Marcus region increases [22]. Using size-quantized semiconductor particles increases the photo efficiency of systems in which the rate-limiting step is charge transfer. Mill and Hunte [23] reported that because the absorption edge blue shifts with decreasing particle size, the redox potentials of the photo generated electrons and holes in quantized semiconductor particles increased. In other words, quantized particles show higher photo activity than macro crystalline semiconductor particles.

## CONCLUSIONS

It is observed that a novel technique developed for the fabrication of Dye Sensitized Solar Cell (DSSC). It may be conclude that with increase in frequency the resistance of the cell decreases. It nearly equal to zero and tends to zero also. With increase in frequency the electron emission from the dye increases. More number of electrons comes from valence band to conduction band so the potential difference between the working electrode and the counter electrode increases and hence energy conversion increases. With a certain frequency, the resistance is very low and maximum number of electron emitted at that frequency is known as maximum operating frequency. Beyond it, there will be no emission of electron with increase in frequency. Here dyes are also important factors for increasing energy conversion efficiency. In this study we observed that henna and pomegranate dyes have better energy conversion efficiency as compared to beet root dye.

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