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# Profilometry Analysis of Flourine Doped Tin Oxide (FTO) Film and Mesoporous (M-$TiO_2$) Film Using Organic Dye from Senna Plant as a Photosensitizer

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## Abstract

This work examines the profilometry characterization of fluorine doped tin oxide (FTO) film and mesoporous (M-$TiO_2$) film using organic dye from Senna plant as photosensitizer. Fluorine doped tin oxide (FTO) film was grown on the substrate using chemical vapour deposition method. Tin-iv-chloride (60%) and hydrofluoric acid (40%) were deposited to the substrate as precursors using two set of bubblers and Nitrogen gas was supply to the same pipe carrying the precursors as a carrier gas. The whole set up was connected to air compressor, which pumps air to the system. The deposited precursors (FTO) were later sintered at 550°C. The first flow meter deposition time is 6 minutes and the flow rate is one (1) litre per minute. The second flow meter connected to the system measured the volume of air that flows into the pipe per time. The grown fluorine doped tin oxide (FTO) film was characterized to find out the thickness and area of the spectra grown on the substrate.

**Keywords:** Profilometry; Doped; Precursors; Compressor; Substrate

## Introduction

Dye –sensitization solar cell is a device that converts visible light into electricity based on the photosensitization of wide band gap metal oxide-semiconductors using dye molecules [1]. Dye-sensitized solar cell operation is based on a semiconductor formed between a photon-sensitized anode and an electrolyte, a photo electrochemical system [2]. Dye sensitized solar cells are environmentally friendly, biodegradable, non-toxic and can be developed using simple methods and used without any purification [3,4]. Dye molecules rather than an inorganic semiconductor material are responsible for light absorption.

The main idea in dye-sensitized solar cell (DSSC) development process is that electron transport takes place in a porous $TiO_2$ structure, while hole transport occurs in a liquid redox electrolyte [5-10], thereby resembling natural light harvesting procedures in photosynthesis. This enables the combination of dye sensitizers with semiconductors [11-14]. Natural pigments that are freely available in plant leaves, flowers, and fruits of natural plants includes chlorophyll, carotene and cyanine are all useful in this absorption and transport mechanism of Dye-sensitized solar cell(DSSC) [15-22].

The range of absorption spectrum of a semiconductor material is important for its uses. The useful semiconductors for photo catalysis have a band gap values comparable to photons energy of visible light, having a value below 3.5 eV [6,7]. The band gap values for the three crystalline forms of $TiO_2$; anatase, rutile and brookite are 3.20 eV (tetragonal), 3.02 eV (tetragonal) and 2.96 eV (orthorhombic) respectively [3-5].

Different doping methods have been adopted to modify the electrical characteristics of $TiO_2$ nanoparticles in order to achieve new or improved catalytic characteristics and other chemical and physical properties [8,9,11,13]. The aim of this research is to show the profilometry analysis of fluorine doped tin oxide (FTO) film and Mesoporous film (M-$TiO_2$) using organic dye from Senna Plant as photosensitizer. The scope of this research covered the Preparation of Fluorine Doped Tin Oxide (FTO) Film and Mesoporous film (M-$TiO_2$), photosensitization of the prepared films using organic dye prepared from *Occidentalis* (Senna Plant), characterization result of this research will be use as part of the requirement for the fabrication of prototype of Dye-sensitized solar cell (DSSC) at the end of this research.

## Materials and Methods

### Preparation of Fluorine Doped Tin Oxide (FTO) Film

The soda lime glass was carefully cleaned using wool and piranha solution to remove any dirty particles on the soda lime glass. Fluorine doped Tin oxide thin films were deposited on the soda lime glass substrates (Axion Medicals UF) using chemical vapour deposition (CVD) Method. 60% of Tin (IV) Chloride ($SnCl_4$) and 40% of hydrofluoric acid were deposited on the cleaned soda lime glass as precursors and Nitrogen gas was used as a carrier gas.

Nitrogen gas was fed from a cylinder through a pressure regulator (Glook scientific) set at 0.5 Bars and then through a mass flow controller (Alicat Scientific). The flow rate is set at one (1) litre per minute and then through a bubbler containing $SnCl_4$ (anhydrous).

The two gas streams converge on the substrate maintained at 550°C by means of a thermocouple and temperature controller (Rex C-900). A chemical reaction takes place leading to the deposition of a transparent and conductive fluorine doped tin oxide (FTO) thin film. The deposition time is varied between one minute and 5 minutes to generate films of transparent and conductive qualities.

### Deposition of Titanium Dioxide Paste ($TiO_2$) on the Soda Lime Glass (Formation of Mesoporous Layer of DSSC)

Mesoporous $TiO_2$ was deposited on the same substrate containing fluorine doped tin oxide (FTO) and blocking layer grown film. Firstly, in order to form mesoporous layer on the substrate, the portion to be coated was placed under the square dimension marked on the screen cap of the printer. Thereafter, the substrate was firmly held with masking tape. The printing cover of the screen printer (cap) was gently released to make contact with the surface of the substrate placed inside the square dimension unit marked under the printers cap. The printing was performed on the same side of the substrate where the film has already grown by robbing small quantity of $TiO_2$ on the positioned substrate via the printers mesoporous cap. The substrates coated with $TiO_2$ was printed or grown for the purpose of developing dye sensitized solar cell. After screen printing $TiO_2$ on the substrate, the substrate was dried and annealed at 550°C for the period of 30 minutes to allow the $TiO_2$ settle well and absorbed inside the substrate. After annealing, $TiO_2$ was deposited for the second time using screen printing method to increase the thickness of the cell from 7 microns to 14 microns. The $TiO_2$ used was purchased from Solaronix Shop Abuja, $TiO_2$-Titanium dioxide z/sp 100g.

### Preparation of natural dyes

The leaves of *Senna Occidentalis* (Senna Plant) were provided and name by a Botanist. The first step was to measure five grams (5 g) of each leave using weighing balance. Mixture of water and methanol 60ml (50:50 ratios) was measured using cylinder and grinded using electronics grinder. The five (5 g) of leave and 60ml of mixed measured solvent was poured inside a grinder. The 5 g of leave and 60ml of solvent (50:50 ratio of water and methanol) was grinded for five (5) minutes. After grinding, the solution was filtered and the dye separated and poured into a cylinder. The filtrate was further filtered by placing the solution on centrifuge machine. The centrifuge machine was allowed to centrifuge for 3 minutes. At the end of this operation, the natural dye from the leaf was on top of the cylinder while the waste product clustered at the bottom of the cylinder. The filtered dye was carefully transferred to a small container that has cover. The dye was packed in a dark cupboard to avoid absorption of incident photon in open space.

### Sensitization of nano crystalline fluorine doped tin oxide (FTO) film using natural dye

The prepared natural dye from *Occidentalis* (Senna Plant) was used to sensitize the fluorine doped tin oxide (FTO) substrate. Fluorine doped tin oxide (FTO) film was soaked inside the prepared natural dye for the period of twelve (12 hours).After soaking the nanocrystalline substrate inside the dye, the soaked substrate was washed using the prepared solvent (50:50 ratio of water and methanol)

The flowchart for the fabrication of Dye-sensitized solar cell (DSSC) development is shown in **Figure 1.**

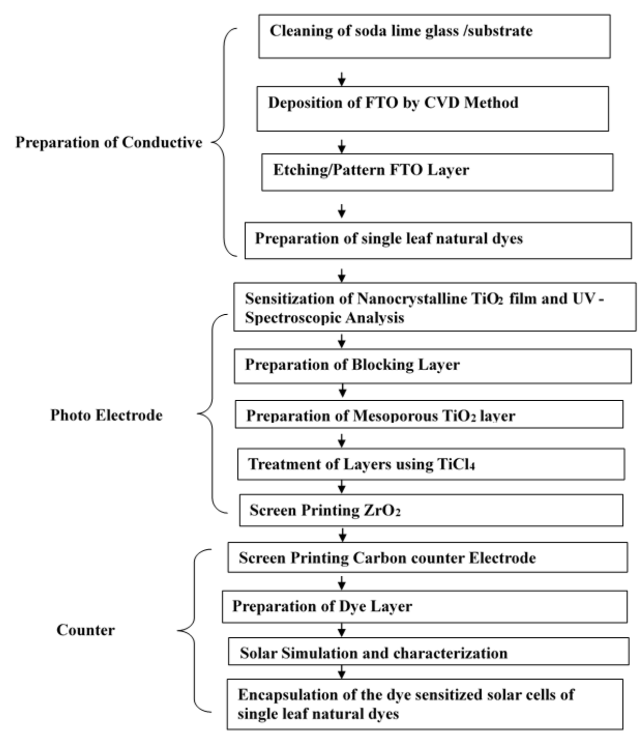


**Figure 1:** Flowchart for the fabrication of Dye-sensitized solar cell (DSSC).

## Results and Discussion

It was noticed that as the wavelength increases the absorbance decrease in **Figure 2**. The liquid samples recorded the highest absorbance and they absorb more cells compared to the solid samples. The liquid sample recorded absorbance above 2.0 A.U. while the solid sample as an absorbance within 0.1 – 0.27. From the plot, the solid sample absorbance will be suitable for solar application while the liquid sample will be for photovoltaic application.

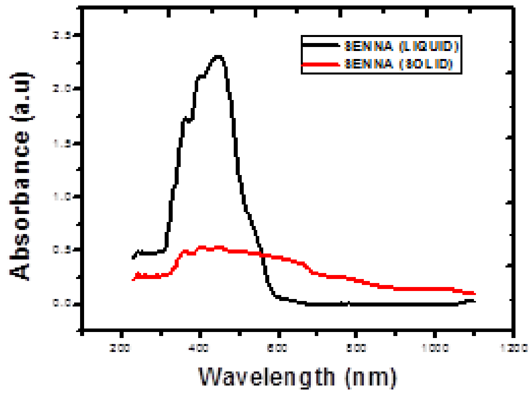


**Figure 2:** The plot of absorbance as a function of wavelength for Senna (liquid and solid sample).

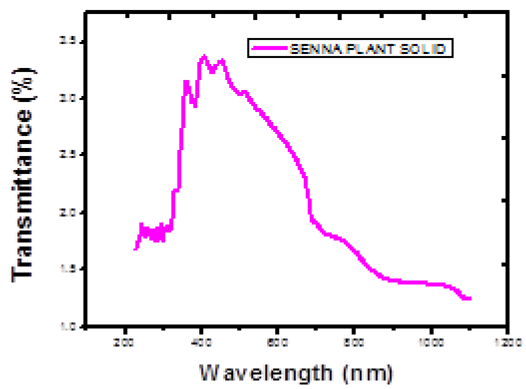


**Figure 3:** The plot transmittance (%) as a function wavelength for Senna plant.

**Figure 3** shows that the transmittance increases as the wavelength of incident radiation increases. The transmittance is 55% in the infrared region and 45% in the ultraviolet region. Films of low transmittance in the infrared region is used in the mass production of solar cells and for the fabrication of solar panel while films with high transmittance in the ultraviolet region is useful in photosynthetic coatings because they exhibit selective transmittance of photosynthetic active radiation (PAR) and also used as reflector and dielectric filter.

The band gap energy as obtained for Senna Plant synthesized on fluorine doped tin oxide (FTO) is 3.49 eV. From **Figure 4**, it was observed that the absorption coefficient squared increases exponentially with photon energy. The range of the band gap energy makes the material useful for fabrication of blue and green light emitting devices, photocell window layer and light emitting laser diode

**Figure 5** shows the absorption coefficient spectra of Senna films sharp edges at the lower energies. This is because these light energies below the band gap (3.0eV and above) do not have sufficient energy to excite an electron into the conduction band from the valence band. Consequently, the light was not absorbed. But, as the photon energy increased sufficiently to about 3.25eV, the absorption coefficient increases with the photon energy. Materials with higher absorption coefficients just like Senna plant more readily absorbs photons which excite electrons into the conduction band.

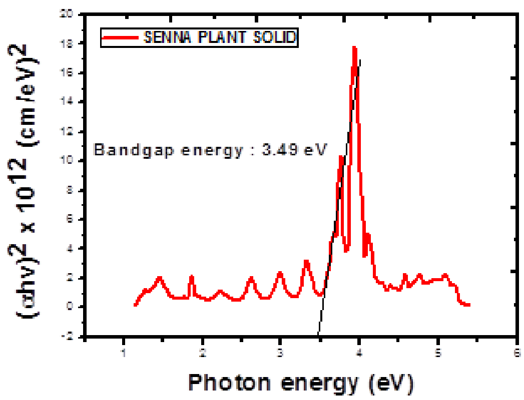


**Figure 4:** The plot of absorption coefficient square as a function of photon energy for Senna plant.

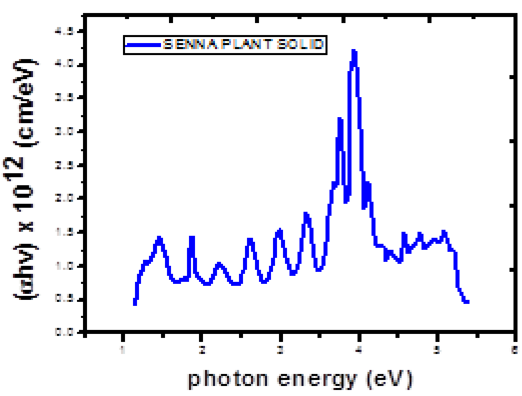


**Figure 5:** The plot of absorption coefficient as a function of photon energy for Senna plant solid.

## Performances of $TiO_2$-based Dye-sensitized solar cell (DSSCs) using inexpensive natural dye extracts

The current density plots as a function of the voltage for the Dye-sensitized solar cell (DSSCs) for Senna plant natural dye is shown in **Figures 6a and 6b.**

The Senna Plant natural dye extracts revealed good absorbance. The efficiency of a solar cell and incident power converted to electricity was calculated using equations 1 and 2.

The Dye-sensitized solar cell (DSSCs) fabricated using these natural dyes gave overall photocurrent conversion efficiencies of η 0.028%. One of the greatest challenges to $TiO_2$ Dye-sensitized solar cell (DSSCs) is its ability to strongly absorb dye molecules.

The efficiency of a solar cell is determined as the fraction of incident power which is converted to electricity and defined by the given equations

$$P_{max} = V_{OC} I_{SC} FF \quad (1)$$

$$\eta = \frac{V_{OC} I_{SC} FF}{P_{in}} \quad (2)$$

$$FF = \frac{1_m V_m}{V_{OC} I_{SC}} \quad (3)$$

Where;

VOC is the open-circuit voltage, ISC is the short-circuit current; FF is the fill factor and η is the efficiency and Pin is incident power. The resulting graph of current/voltage and voltage power for Senna plant are shown in **Figures 6a and 6b** respectively.

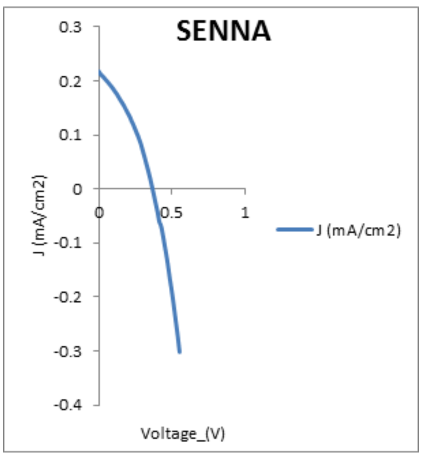


**Figure 6 (a):** Graph of Current vs. Voltage for senna plant.

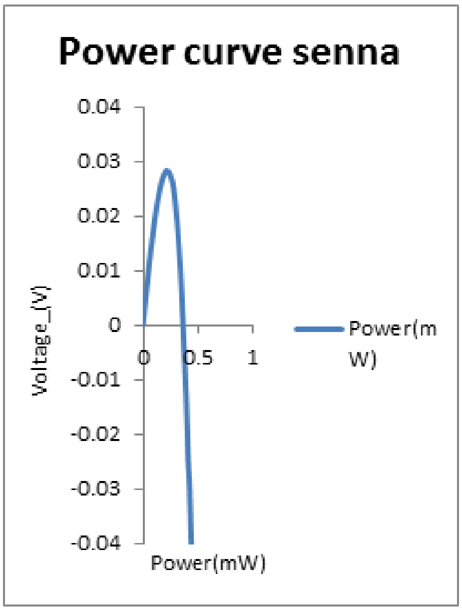


**Figure 6 (b):** Graph of Voltage power for Senna Plant.

**Four Point Probe Analysis**

Four point probes is a simple apparatus for measuring the resistivity of semiconductors samples.

Four point probe is also called 4-wire sensing or four-terminal sensing which is an electrical impedance measuring technique that uses separate parts of current carrying and voltage sensing electrodes to make more accurate measurements than the traditional two terminal (2T) sensing . It is used in some ohm meters and impedance analyzers and in precision wiring configuration for strain gauges and resistance thermometers and to measure sheet resistance of thin films. The key advantage of four point probes is that the separation of current and voltage electrodes eliminates the impedance contribution of the wiring and contact resistance.

The electrical properties of the films were investigated using a four point probes technique. The arrangement was made in such a way that the voltage across the transverse distance of the films and the corresponding values of the current were measured using silver paste to ensure good ohmic contact to the film. The values of the resistance for the films deposited were investigated. The four point probes were connected to a current supply and the inner probes to a volts meter. As current flows between the outer probes, the voltage drop across the probes is measured. If the spacing between the points is constant, and the conducting film thickness is less than 40% of the spacing, and the edges of the film are more than 43 times the spacing distance from the measurement point, the average resistance of the film or the sheet resistance is given by equation 2.

## Profilometry Result

A solar simulator is a device that provides illumination approximating natural sunlight. A four Point Probes was used to test whether the fluorine doped tin oxide (FTO) Film is working or not. The purpose of the solar simulator is to provide a controllable indoor test facility under laboratory conditions, used for the testing of solar cells, sun screen, plastics, and other materials and devices. Light sources developed and used to simulate solar radiation are called solar simulators. There are several types of solar simulators with different spectra and irradiance distributions. The solar simulation spectrum is further specified via the integrated irradiance across several wavelength intervals see **(Figures 7–11)**. Each Figure shows D-$TiO_2$ for 5 cycles with each graph appearing to conform with the respective cycle.

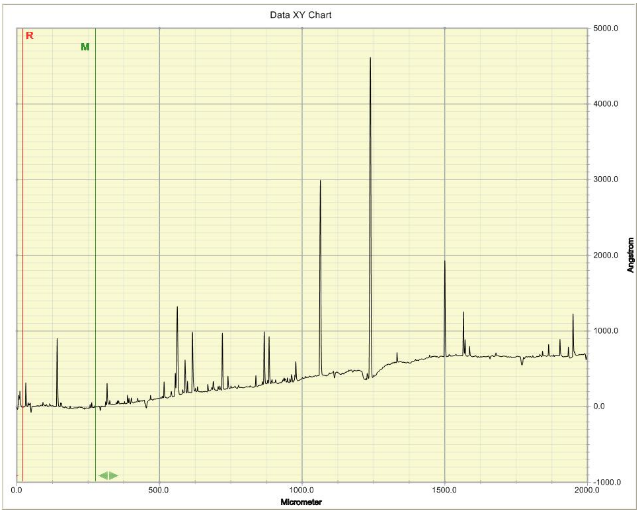


**Figure 7:** Graph Showing D-$TiO_2$-1-cycle.

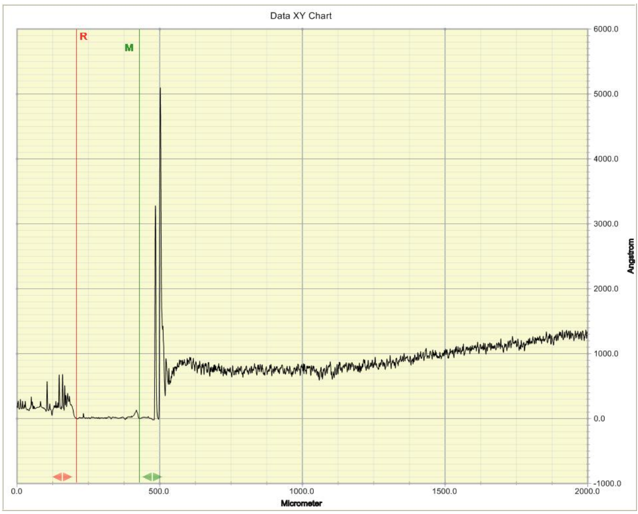


**Figure 8:** Graph Showing D-$TiO_2$-2-Cycles.

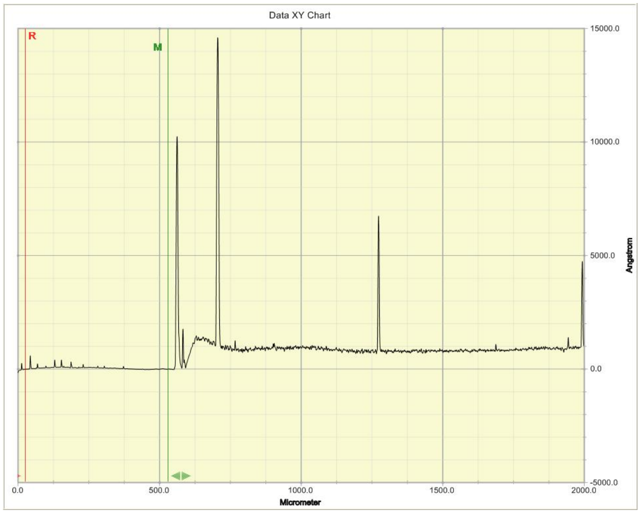


**Figure 9:** Graph Showing D-$TiO_2$-3-cycles.

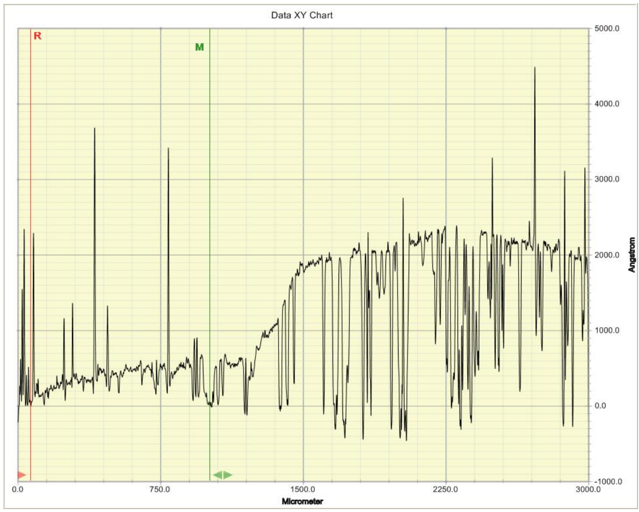


**Figure 10:** Graph Showing D-$TiO_2$-4-cycles.

The Dye-sensitized solar cell (DSSC) fabricated from fluorine doped tin oxide (FTO) Film and mesoporous films showed 1.7 times of photovoltaic current than those from the nanocrystalline films in the same thickness. It is deduced that the high current is caused by the efficient transport of electrons due to far less grain boundaries of fluorine doped tin oxide (FTO) Film and the mesoporous $TiO_2$ structure, and by the fast diffusion of electrolytes with the high uniformity in the mesopore size.

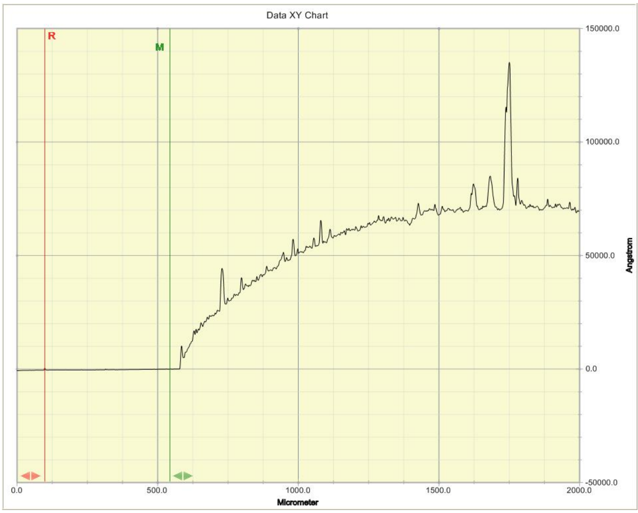


**Figure 11:** Graph Showing D-$TiO_2$-5-cycles.

## Conclusion

It was seen from the characterization that the optical properties of the film were determined. The solar simulation of Dye-sensitized solar cell (DSSCs) developed in this work show the following electrical characteristics (Isc) = 0.217 mA, (Voc) = 0.362 V, FF (%) = 35.99 %, η (%) = 0.028% and MPPT (mW) = 0.028 mW for senna dyed $TiO_2$. The optical properties of these films and electrical characteristics made the material to have wide range of applications especially in optical and electronic systems. The profilometry analysis shows that fluorine doped tin oxide (FTO) Film and the mesoporous $TiO_2$ structure have 1.7 times of photovoltaic current than those from the nanocrystalline films in the same thickness.

It was found out that the liquid samples recorded the highest absorbance and thus absorbing more cells when compared to the solid samples. The transmittance was found to increase as the wavelength of the incident radiation increases. The absorption coefficients show sharp edges at the lower energies.

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