# SnO<sub>2</sub> NANOFIBERS PHOTOANODE FOR ENHANCING THE PHOTOVOLTAIC PROPERTIES OF NATURAL DYE SENSITIZED SOLAR CELLS

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## Abstract

Tin oxide  $(SnO_2)$  nanofibers were successfully prepared by electrospinning homoheneous viscous solution of tin acetate in polyvinyl alcohol (PVA). The electrospun nanofibers are calcined at 600° C for 2 h. Structural, diameter in size and morphology of heat-treated  $SnO_2$  nanofibers were investigated in detail by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and Atomic Force Microscope (AFM). The optical properties of  $SnO_2$  nanofiber film was also studied by UV-Vis spectroscopy. The nanofiber was sensitized with natural dye and assembled into a DSSC. High performance carbon electrode was prepared onto ITO substrate and used as counter electrode. I-V characteristic was measured and conversion efficiency ( $\eta_{COn}$ ) and fill factor (FF) were determined.SnO<sub>2</sub> thin film will be deposited onto ITO (Indium doped tin oxide) coated glass substrate by spin coating technique. After that, the solar cell performance of DSSC with nanofibers will be measured and compared to those of DSSC with SnO<sub>2</sub> thin film. According to the I-V measurement, the efficiency of SnO<sub>2</sub> nanofibers based DSSC was greater than that of SnO<sub>2</sub> thin film based DSSC. The SnO<sub>2</sub> nanofibers based DSSC of TCD 12 cm gave the best efficiency and it was 2.71 %. The experimental findings resulted from this research work can be of great help for low-cost and Eco-friendly DSSC application.

Keywords: Tin acetate, polyvinyl alcohol, Nanofibers, TCD, Electrospinning

## Introduction

Over billions of years, Mother Nature has been converting light from the sun into energy via photosynthesis. Sunlight is the most abundant and sustainable energy source that is free.[Hong Juan Sun et al (2011), Jie GAO Et Al (2013)]. The Earth receives energy from the sun at the rate of  $\sim 12 \times 1017$  J s-1. This has exceeded the yearly worldwide energy consumption rate of  $\sim 1.5 \times 1013$  J s-1. Therefore, it is a challenge to devise an approach for the effective capture and storage of solar energy for our consumption since fossil fuels such as oil and gas will be depleted in the years to come. In order to imitate the photosynthesis process, Gratzel and coworkers have developed dye-sensitized solar cells (DSSCs) based on the similar working mechanism [HATEM S. EL-GHAMRI et al (2014), T.F. Stoica, T.A. Stoica (2000)]. There are three generation in solar cell and the first and second generations for solar cells are mainly constructed from semiconductors. Among of them, dye sensitized solar cells (DSSCs) have been widely explored as potential alternatives to conventional silicon photovoltaic (PV) devices due to their low cost, abundance of raw material, facile fabrication process and overall good photovoltaic performance [Jiawei Gong, HuiQiao (2015)]. Compared to typical semiconductors used in p-n junction solar cells, the materials employed in a DSSC photoanode may have lower purities, there by leading to lower production costs [Sofyan A. Taya et al (2013)].

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The modern version of a dye solar cell, also known as the Grätzel cell, was originally coinvented in 1988 by Brian O'Regan and Michael Grätzel at UC Berkeley and this work was later developed by the aforementioned scientists at the École Polytechnique Fédérale de Lausanne until the publication of the first high efficiency DSSC in 1991. Michael Grätzel has been awarded the 2010 Millennium Technology Prize for this invention [Rajesh Vasita, Dhirendra S Katti (2006), S.S. Dissanayake et al (2016)].

Tin oxide (SnO<sub>2</sub>) is one of the most intriguing materials to be investigated today, This is because tin dioxide isa well-known n-type semiconductor with a wide band gap of 3.6-3.8 eV, and for its potential application in transparent conductive electrode for solar cells a gas sensing material for gas sensors devices, transparent conducting electrodes, photochemical and photoconductive devices in liquid crystal display , gas discharge display, lithium-ionbatteries, etc[Ganesh E Patil (2012)]. Many processes have been developed to the synthesis of SnO2 nanostructures, e.g., spray pyrolysis, hydrothermal methods, chemical vapor deposition, thermal evaporation of oxide powders and sol–gel method [Yanika DiahImawanti (2017),M. T. Raposo (2012)]. Its outstanding electrical and optical properties enable applications in solar cells[Sofyan A. Taya et al (2013), [S.S. Dissanayake et al (2016)].

The DSSCs based on  $\text{SnO}_2$  photoanode materials exhibit relatively low open circuit voltages because of their high recombination kinetics with electrolytes. In addition, they reduce the conversion efficiency [Suriati Suhaimi1 et al (2015)].  $\text{SnO}_2$  with various morphologies of nanograin, nanowires, and nanoparticles have been investigated to increase the photoconversion efficiency of  $\text{SnO}_2$  based DSSCs. The high recombination rate and poor electron transfer efficiency in  $\text{SnO}_2$ -based DSSCs are still major challenges for increasing the energy conversion efficiency [Zhengtao Zhu et al (2010)].

## **Experimental Procedure**

#### Sample Preparation of SnO<sub>2</sub>Nanopowder

In this research, Stannous chloride dehydrate (SnCl<sub>2</sub>.2H<sub>2</sub>O) was used as starting materials. Distilled water was used as a solvent. SnO<sub>2</sub>nanopowders have been prepared by using precipitation method. SnO<sub>2</sub>nanopowders were prepared by means of dissolving of 2 g (0.1 M) stannous chloride dehydrate (SnCl<sub>2</sub>.2H<sub>2</sub>O) in 100 ml distilled water. 10% ammonium hydroxide was slowly added to the solution for complete precipitation of tin hydroxide in the pH range 7.5 - 9. Then the solution was put into thermostat water bath, in which the temperature was kept about 80 °C, for 15 minutes until white depositions came out. Then the precipitate was centrifuged and washed several time (pH =7) with distilled water to reduce the amount of ammonium chloride. The resulting gels was filtered and dried at 100 °C for 3 h. The obtained powder was collected and grounded in an agate mortar and it is referred "as – prepared". The color of the as-prepared sample is gray. Finally the as-prepared sample was heated in a muffle furnace at 650 °C for 6 h in an atmosphere, then the color turned into white. Field emission scanning electron microscopy (FESEM) was employed for morphological study using ZIESS (MERLIN).

#### **Cleaning of the Substrate**

In the present study ITO/glass, cut into  $1 \times 1$  cm square plates have been used as substrates. Prior to deposition the substrates are subjected to the following cleaning process to remove the unwanted impurities normally present on the surfaces of the glass plates exposed to the atmosphere.

- 1. The glass substrates are washed in soap solution by scrubbing the surfaces with a cotton swab dipped in liquid soap.
- 2. The glass slides are then rinsed thoroughly in deionized water to remove any trace of the soap solution left on the surface followed by acetone rinsing.
- 3. Then the slides are soaked in HCl acid and kept in room temperature for about two hours.
- 4. Finally, the substrates are rinsed thoroughly in deionized water and dried at 100°C for one hour in an oven.

## Preparation of SnO<sub>2</sub> Paste and Spin Coating of Tin Oxide Films for Photo Electrodes

The paste has been prepared by 2 g of  $\text{SnO}_2$  powder was dissolved into the 100 ml of ethanol. The mixture was stirred by magnetic stirrer for 2 h. The solution was then ultrasonicated for 4 h to reduce particle size. After sonication, the paste was ready to use as spinning paste. After that the film were deposited on ITO/glass substrate. After spin coating at room temperature, and at a speed of 3000 rpm for 30 s, the coated films were dried 15 minutes at 150 °C. The formation of ITO films by removing the organic component followed by structural rearrangement was performed by annealing the coated films for one hour at the temperature of 400 °C. The structural properties of the films were measured by field emission scanning electron microscope (FESEM).

## Low Cost Home-made Electrospinning Set-up for SnO<sub>2</sub> Nanofiber

Electrospinning is a very simple technique that can be used to produce nanofibers under the influence of a high electric field. This home-made electrospinning set-up consists three components. They are home-made high voltage power supply, syringe (emitter) and collector plate. The positive terminal of the DC source was connected with a needle and the Al-substrate (collector) was connected with the negative terminal to ground it. When the voltage was applied a stream of solution came out through the needle which was subdivided into a number of nano to submicron sized jets and were deposited in the form of nanofibers on the collector pate.

## 4.4 Design and Construction of Home-made High Voltage Power Supply

The power supply is constructed to obtain large output voltages (dc), at low currents. The output can be adjusted using the potentiometer on the outside of the box. Here are the important specifications.

Output voltage range: 1000 – 20000 V DC Maximum output current: 20 mA Input voltage: 210 - 250 V AC Input current: 0.5-1 Amperes. Average power of device: 40 – 60 Watts

The power supply is plugged in to the ac-mains to power on. Make sure that before powering up, the potentiometer is tuned to its lowest value, this must be secured in order to minimize the risk of injury, as high voltages can be very dangerous. To use the power supply to get high voltages, connect probes to the positive and negative crocodile clips provided. The potential difference will exist between these two crocodile clips. They can be connected in necessary places in various apparatuses used in high voltage experiments. There is no display to show the output voltages in the power supply itself. This is because common voltmeters available in labs cannot measure voltages more than 1000-1500 volts. So, to measure the output voltages, the user will have to use a variable resistor (EMCOV1G). This device is basically a high voltage step-down resistor, which allows the user to see output voltages across its terminals that are stepped down by a factor of 1000, e.g., if a multimeter joined to the output of the EMCO device shows 20 V, it means that there is actually a voltage of 20000 V at the output of the high voltage power supply. So, simply connect the resistor to one of the high voltage outputs, and then observe the voltage on a multimeter. To do so, connect the high voltage input wire of the ECOM device to the high voltage output, and connect the mutlimeter between the ground of the power supply and the positive output of the ECOM device. This home-made HV power supply was composed of five sections and the block diagram are shown in figure 1. The experimental detail for each section are as follow:

**Step-down Transformer:** This is basically a transformer that connects to the ac –main, steps the voltage down, and gives an output voltage about 40 V. The core is made up of f-type plates. The primary contains about 1000 turns of insulated copper wire. The secondary contains about 200 turns of copper wire. The secondary wire is thicker because more current will flow through the secondary than at the primary, as this is a step-down transformer.

**Voltage regulator circuit:** This is a simple circuit that as the main source of power for the flyback transformer and its driver. The circuit contains 3 outputs, one 5 V, one 12 V, and a regulated output, between 1-30 V dc. The output voltage of the regulated output can be changed from the variable resistor, and this is the main voltage that is given to the input of the flyback transformer, and hence it controls the output voltage.

**Flyback-transformer driver circuit:** This is a circuit that basically drives the flyback transformer. The main function of this circuit is to simulate a pulsed (square-wave), high frequency input for the flyback transformer, which then allows it to work. The square wave generated typically has a frequency between 25-100 kHz, in this case it was about 26 kHz, but the exact value can vary from one flyback transformer to another. The circuit consists a 55-timer, which is put in the astable mode, this generates a square wave. The output of the 555-timer is fed into the input of a hex-inverter buffer. The output of this buffer acts as an open-collector, a sink for the 12 V dc from the regulated circuit. Periodic transitions of the output of the buffer from open, to a short sink, allow it to switch the transistor on and off. The transistor being switched on and off is an n-channel mosfet, in our case it is an IRF-540. We chose this particular transistor because of its low drain-source resistance, so it heats up less. The transistor is connected in a heavy-side driver configuration, in which the primary of the flyback transformer is placed between the drain and the regulated input from the voltage regulator circuit.

**Flyback-transformer:** A flyback transformer is a very interesting transformer. Normal transformers, simply transformer energy from their inputs to their outputs. Flyback transformers, however, not only transfer energy to their secondary, but also they store energy for a considerable amount of time. In fact, flyback transformers act as pure inductors during half a cycle, and then they act as a pure transformer in the other half. This behavior is made possible by

an air gap in the ferrite core. This air gap increases the reluctance of the core, which thereby increases its ability to store magnetic energy. Flyback transformers are high frequency transformers, and their low output power and small size make them very useful in generating high output voltages, at relatively low currents. Their main applications are in televisions, monitors, and high voltage power supplies, like this one. The circuit diagram and the photo of homemade high voltage power supply were shown in figure 2 and 3.



Figure 1 The block diagram for home-made high voltage power supply



Figure 2 The circuit diagram of HV power supply



Figure 3 Home-made high voltage power supply

#### Sample Preparation of SnO<sub>2</sub> Nanofiber

In this study, the flow chart of experimental procedure is shown in figure 4.4. Tin dioxide  $(SnO_2)$ , Poly Vinyl Alcohol (PVA) and distilled water were chosen as the starting chemicals and solvent. SnO<sub>2</sub> nanofibers were prepared by sol-gel process. 1 g of SnO<sub>2</sub> was dissolved in 2 ml of acetic acid with constant stirring for 10 min to obtain a clear solution of tin acetate in the pH range 1-2. This solution was mixed with 4 ml of 10 % PVA solution. The solution was stirred for 4 h by a magnetic stirrer and a viscous sol-gel was obtained. Nearly 5 ml of the viscous solution was taken in a 20 ml syringe. The distance of 12 cm was maintained between the collector plate and the tip of the needle. Al-substrate was then struck on the collector. When the voltage was applied a stream of solution came out through the needle which was subdivided into a number of nano to submicron sized jets and were deposited in the form of nanofibers on the collector pate. The green nanofibers were calcined at 600 °C for 2 h. A sketch and photo electronspinning set up is shown in figure 4 and figure 5. The electrospinning conditions are mentioned in table 1. The obtained sample was characterized by X-ray powder diffraction (XRD) using (Rigaku RINT 2000). The morphology of the fibers was measured by field emission scanning electron microscope (FESEM) and atomic force microscope (AFM).



Figure 4 Schematic drawing (a) and photo(b) of the electrospinning process set up

Syringe capacity	20 ml
Tip to collector distance (TCD)	12 cm
Voltage	20 kV
Annealing temperature	600 °C
Annealing time	2 h

Table 1 The elctrospinning conditions of SnO<sub>2</sub> nanofibers

## **Preparation of Natural Dye-Sensitizer**

The grass from 16° 52' 59.69" N 97° 37' 51.02" E was used as a dye in this work. Firstly, the grass was washed with deionized water and dried in dark place at room temperature for about 3 months. After drying, the grass was crushed into small pieces using mortar. Then, dried small grass parts were put into thistle of soxhlet extractor and ethanol was used as solvent. Temperature of the instrument was maintained well under boiling point of the ethanol. Several cycles of solvent were run so as to extract all the compounds from plant parts. Next, the ethanol from dye solution was removed by distillation. Finally, the pure natural dye solution (pH=6.67) was obtained and it was ready to be used as a natural dyes sensitizers in DSSCs.

#### **DSSC** Assembling

The cathode electrode and the anode electrode were put together, overlapping each other, and a space at the end of each electrode was made. Next, both electrodes were fasten using the double clip. Three drops of iodide solution were added at the end of the electrode and the solutions were spread over the entire electrode. Then, the remaining iodide solution were wiped off using cotton swab soaked with alcohol. After that, a tester with crocodile clip were attached at both ends of the electrode.

## **Results and Discussion**

## X-ray Diffraction Methods on Structural Characterization of SnO<sub>2</sub>nanofibers

According to the XRD analysis, pure tin oxide nanofibers were matched with standard library of PDF 77-0447 cassiterite tin oxide. The X-ray diffraction (XRD) pattern of SnO<sub>2</sub>nanofibers from SnCl<sub>2</sub>.2H<sub>2</sub>O is shown on figure 5. The peaks at 20 values of 26.588°, 33.848°, 37.878° and 51.720° can be associated with (110), (101), (200) and (211) respectively. All of the peaks can be indexed to be pure tin oxide structure of tetragonal due to the lattice parameters agreement with the literature. The average crystallite size is 27.49 nm.



Figure 5 XRDdiffractorgram of tin oxide nanofibers

Table 2 Averag	e crystallite size	of pure SnC	<b>D</b> <sub>2</sub> nanofibers	from SnCl	2.2H <sub>2</sub> O for	all identified
peaks a	at 650 °C					

No.	Peak	FWHM (deg)	2-Theta(deg)	Crystallite size (nm)
1	(1 1 0)	0.48	26.58	16.90
2	(1 0 1)	0.27	33.84	33.62
3	(2 0 0)	0.27	37.87	34.00
4	(2 1 1)	0.34	51.72	25.44
	27.49			

Table 3 Lattice parameters (a & c) and lattice distortion c/a for SnO<sub>2</sub>nanofibers

	Lattic pa			
	a( A°)	c(A°)	C/ a	
SnCl <sub>2</sub> .2H <sub>2</sub> O	4.739	3.193	0.673	
Standard	4.737	3.185	0.672	

#### **Surface Morphology**

The SnO<sub>2</sub> powder was obtained from directed precipitation method. Figure 6 (a) shows the FESEM analysis of the SnO<sub>2</sub> powder. The grain sizes were calculated by using well known bar code system. Bar code size was 1  $\mu$ m. According to the calculation the grain size is 80 nm – 120 nm. The powders morphology was spherical in shape. The FESEM images of SnO<sub>2</sub> films for 400 °C was shown in Figure 6 (b). According to the FESEM images the sizes of the grain are non-uniform. The various sizes of particles spread out on the surface of the film. The average grain sizes of SnO<sub>2</sub> films for all temperature are 75 nm – 300 nm respectively. The SnO<sub>2</sub> fibers on ITO were carried out to examine by FESEM image. According to the FESEM analysis SnO<sub>2</sub> nanofibers reveal the retention of cylindrical shape but with surface roughness. The average diameter of the SnO<sub>2</sub> nanofibers are 80 – 85 nm. The fibers diameter were also confirmed by AFM analysis. Fiber diameter is estimated from fiber height to avoid tip-convolution effects. According to the line profile, the average fibers diameter are 84 nm – 95 nm.



Figure 6 The FESEM images of  $SnO_2$  (a) nanoparticle (b) film (c) nanofibers  $20\mu m$  (d) nanofibers  $2 \mu m$ .



Figure 7 The amplitude (a), the 3D image (b) and line profile (d) of SnO<sub>2</sub> nanofibers

#### Optical Characterization of SnO<sub>2</sub> nanofibers and natural dye sensitizer

The UV-Vis spectra of SnO<sub>2</sub>nanofibers were recorded with respect to the ITO glass substrate placed in the reference beam using beam spectrometer in the range 190 to 1100 nm. The absorption spectrum of SnO<sub>2</sub> deposited on ITO glass substrate is shown in figure 8 (a). The figure shows high absorption coefficient in the UV region. The resultant values of Eg for SnO2 is found to be about 3.82 eV. The absorption spectrum of grass dye was obtained from UV-Vis spectroscopy. The wavelength range of spectrum between 400 nm and 800 nm. In this absorption spectrum, it was observed that the wide range absorption peaks and all of absorption peaks were within visible region. The strongest absorption peak was found at the wavelength of 671.50 nm and it was contributed that chlorophyll a. In accordance with the Beer-Lambert law that the absorbance value (A) inversely proportional to the value of transmittance (T) and is proportional to the concentration of the solution (C). The maximum absorbance is obtained in this research for 4.00. It shows the greater the concentration of the extract of grass in the solution, the greater the absorbance values and the smaller the transmittance values. Because the chlorophyll content affects the absorbance value of sample's concentration. The absorbance spectrum of extract grass was described in figure 8 (b). Peak absorbance at a wavelength  $\lambda$ = 469 nm with absorbance at 4.00 and  $\lambda$ = 671 nm and with absorbance of 2.82. So the extract dye from the grass has a greater efficiency when applied to dye-sensitized solar cells (DSSC)



Figure 8 The absorbance spectrum of SnO<sub>2</sub> nanofibers (a) and dye solution (b).

## **Photovoltaic Properties**

Figure 9 (a) showed the I-V curves of SnO<sub>2</sub>naofibersphotoelectrodeDSSC and figure 9 (b) showed I-V curve of SnO<sub>2</sub> film based DSSC under illumination. The summarized photvoltaic parameters were listed in Table 4. From the Figure, it was found that the Photovoltaic behaviors of DSSC were quite acceptable, appropriate and suitable for cost effective DSSC with natural dye extract.



Figure 9 I-V curves of  $SnO_2$  nanofiber photoelectrode (a) and film based photoelectrode (b) DSSC solar cell

Table 4	Photovoltaic	parameters	of Sn(	<b>D<sub>2</sub> nanofiber</b>	and	film	photoelectrodes	with	dye
	sensitizer ext	tract							

Sample	Isc(mA)	Jsc(mA/cm2)	Voc(V)	Efficiency (%)	FF	P <sub>m</sub> (mWcm <sup>-2</sup> )
Film	2.79E-02	1.40E-02	0.2240	1.147	0.36	3.199E-3
Nanofiber	4.61E-02	4.60E-02	0.2857	2.715	0.68	1.065E-1

## Conclusion

Dye-sensitized solar cells based on SnO<sub>2</sub>nanofiber and nanopowders were successfully prepared and investigated on their structural, microstructural and light absorption properties. Both SnO<sub>2</sub>nanofibers and SnO<sub>2</sub>nanopowders were deposited onto indium doped tin oxide (ITO) glass substrates by electrospinning and spin coating method. The experimental data resulted from this research gave that both SnO<sub>2</sub>nanofibers and nonopowders were successfully favoured on ITO/glass even though the fabrication technique used in this study was not confused and complicated. . From I-V graphs, the efficiency of nanofibers based DSSC was better than thin films based DSSC. The efficiency of nanofiber based DSSC (TCD 12 cm) 2.715 %. It is expected that both SnO<sub>2</sub> nanofibers based DSSC and SnO<sub>2</sub> thin film based DSSC with grass natural dye are cost effective and Eco friendly DSSC. Moreover, the changing of nanoparticle (0 D) to nanofiber (1 D) is one of the most promising way to enhance the performance of dye sensitized solar cells.

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