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PREPARATION AND CHARACTERIZATION OF SnO_2 NANORODS AT DIFFERENT TEMPERATURES

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Abstract

In this study , tin oxide (SnO_2) nanorods were fabricated onto FTO substrates via chemical bath deposition (CBD) method. SnO_2 nanorods were prepared by immersing SnO_2 seed layer coated FTO glasses into the aqueous solution. After fabrication process, the substrates were annealed at different temperatures and SnO_2 nanorods were formed. And then, SnO_2 nanorods were characterized by X-ray diffraction to observe crystal structure. Rod's diameter and morphological properties at different temperatures were carried out from SEM analysis.SnO_2 nanorods were obtained as an 1D structure. From this experiment, these nanorods would be used to construct the dye-sensitized solar cells (DSSCs) as photoelectrode. Current density –voltage (J-V) characteristics of DSSC with natural dye was measured. From J-V curve, conversion efficiency (η) and fill factor (FF) were evaluated for DSSC.

Keywords; *SnO_2 nanorods ,CBD method , XRD ,SEM , DSSC, J-V Characteristics*

Introduction

Transparent conducting oxide (TCO) substrates have been extensively used in various electronic and optoelectronic applications, such as solar cells , window heaters and liquid crystal devices [Surawut Chuangchote et al,2011]. Most efforts in TCO have Transparent conducting oxide (TCO) substrates have been extensively used in various electronic focused on the oxides of tin , indium and zinc with small amount of other elements as dopants [Surawut Chuangchote et al ,2011]. Nanostructures synthesis with controllable properties is a research desire and still a major challenge for their applications in a wide range[M.A. Batal et al ,2012]. Nano architectures of transition semiconductor oxides are getting immense importance because of their

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excellent properties and potential applications in energy storage devices [M. Zubair Iqbal et al ,2014] .Tin dioxide (SnO_2) is an n-type semiconducting oxide with a wide bandgap (3.6eV) and well known for its potential applications in dye-based solar cells, semiconductor, photoconductors, and gas sensor [Jr H.He et al ,2006].Tin oxide (SnO2) thin film is a wide band gap n-type semiconductor with high simultaneous electrical conductivity and optical transparency in visible region of the spectrum [M.A. Batal et al ,2012]. The properties of SnO_2 materials are strongly dependent on their size and shape, it is obvious that the controlled synthesis of the morphologies of SnO_2 materials is very important for special applications [M. Zubair Iqbal et al ,2014].

Inorganic materials with different morphologies and size can exhibit different properties. Accordingly, various structural and morphological forms of SnO_2 materials have been fabricated over the past several years, including nanowires , nanoribons or nanobelts , nanorods , nanotubes ect [Hyoun Woo Kim et al , 2005]. Several methods and techniques were developed to produce different nanomaterials. Metal oxides having nanostructure forms are of great importance for verity of applications. Among them tin oxide, which has two oxidation states namely stannic (SnO2) and stannous (SnO). Fabrication of SnO_2 nanorods has been accomplished using several vapor deposion techniques, such as chemical bath deposition, thermal evaporation and hydrothermal [O.Lupan et al, 2009]. However, the preparation of SnO2 nanostructures with well-controlled morphology and dimension by facile synthesis is still a challenge [M. Zubair Iqbal ,2014].

In this paper , SnO_2 nanorod on FTO substrates were observed by using chemical bath deposition method. One dimensional (1-D) nanomaterials, such as nanowires, nanorods and nanotubes, are regarded as catalysts for oxidation of organic compounds, solid state sensors, biomedicine, ceramics and transparent conductors.

Experimental Procedure

Fabrication of SnO_2 Nanorod

SnO_2 nanopowder was used as starting material. The well-dissolved precursor solution was firstly prepared by mixing SnO_2 powder (3g) and ethanol solvent (40ml). The mixed solution was stirred with magnetic stirrer for 12h figure 1 and put at 110°C for 1h using water bath to remove water of crystallization, reactive with SnO_2 solution and cooled down at room temperature. Finally, SnO_2 precursor solution was obtained.

The substrates was cleaned in the ultrasonic bath with ethanol and deionized water to remove adsorbed dust and surface contamination.

In this study, FTO glass substrates were used for the growth of SnO_2 seed layer by spin coating technique.

The substrates were placed on fragment adapter and the SnO_2 sol solution was poured onto substrates. The spin speed or rotational speed was set 3000 rpm and spinning time was 30 s figure 2. After spin coating, they were annealing at 500°C, 550°C, 600°C for 1h respectively.

For the growth of SnO_2 nanorod, an aqueous solution of tin chloride dehydrate ($SnCl_2.2H_2O$) was prepared as tin source and hexamethylenetetramine (HTMT) ($C_6H_{12}N_4$) with water was used as oxygen source.

Next, the seed layer coated FTO glasses were tilted against the wall of the aqueous solution of container as shown in figure 3. Then, the containers were heated at 80°C for 5 h. At the end of fabrication process, the substrates were taken out of the solution and rinsed five times with deionized water and dried at room temperature. After these substrates annealed at 240°C for 1 h respectively, SnO_2 nanorod were obtained.

Figure 1. Magnetic Stirrer

Figure 2. Spin Coating Machine

Figure 3. SnO_2seed layer coated FTO glasses immersed into the aqueous solution

Results and Discussion

X- ray Diffraction Analysis

The XRD pattern shown in figure 4 indicates a high purity of SnO_2 powder. Four obvious peaks were formed at (110) , (101) , (200) , and (211) planes .These diffraction peaks indicate a tetragonal structure of SnO_2 with lattic constants of a , b = 4.73 A° and c = 3.18°A that agree with documented values for the SnO_2 crystals. Scherrer's equation was used to estimate the size of SnO_2 crystals . It is stated that the crystallite size $D = \frac{0.89\lambda}{\beta cos\theta}$, where λ is the wavelength for the Cu K_α (= 1.54056A°), β is the broadening at half the maximum intensity (FWHM) expressed in radian , and θ is Bragg's angle. The crystallite size is 74.95 nm for SnO_2 powder based on the (110) peak.

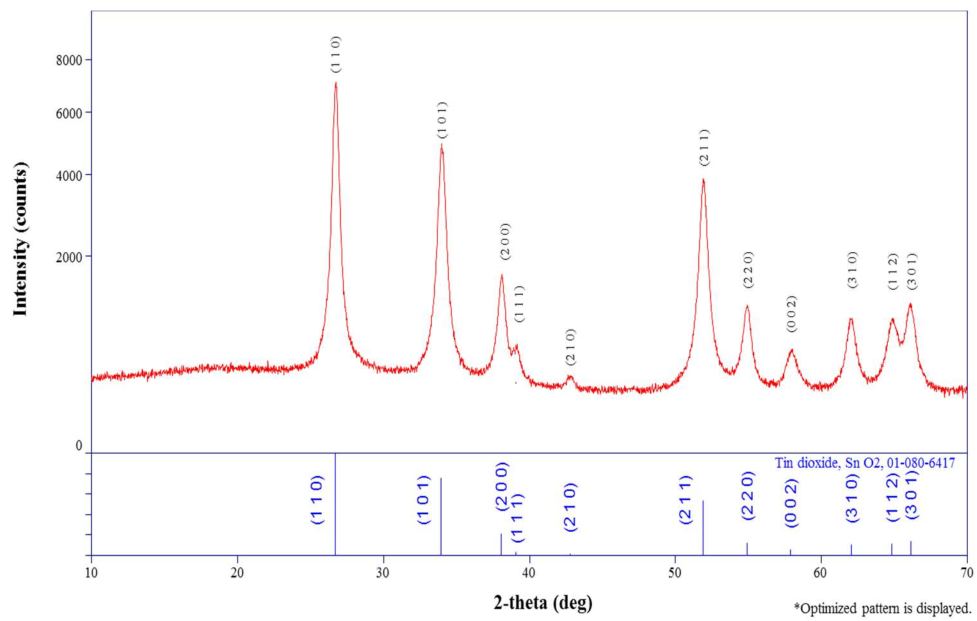

Figure 4. X-ray diffraction pattern of SnO_2 powder

Morphological Characterization

The morphological characterization was analyzed by SEM (Scanning electron Microscope). Figure 5, 6 and 7 showed the SEM image of SnO_2 nanorod at different which a large area of nanorod were formed. The different diameters of SnO_2 was formed between 300-700 nm and length reaches up to around 5µm as shown in Figure 5,6 and 7. The products consists of nanorod as well as nanoparticles. At 550°C , the SnO_2 nanorod were also temperatures were obtained from seed layer coated substrates were tilted against the wall of container. The SnO_2 nanorod showed 1D structure. After calcinations at 500°C, SnO_2 nanorod onto FTO substrate were obtained 1D structure in formed as nearly same size of the product at 600°C , but they were more smooth than the product at 500°C.The SEM images shows that the growth direction of the nanostructure is randomized. In the SEM images, the products with a closer view comprises straight structures.

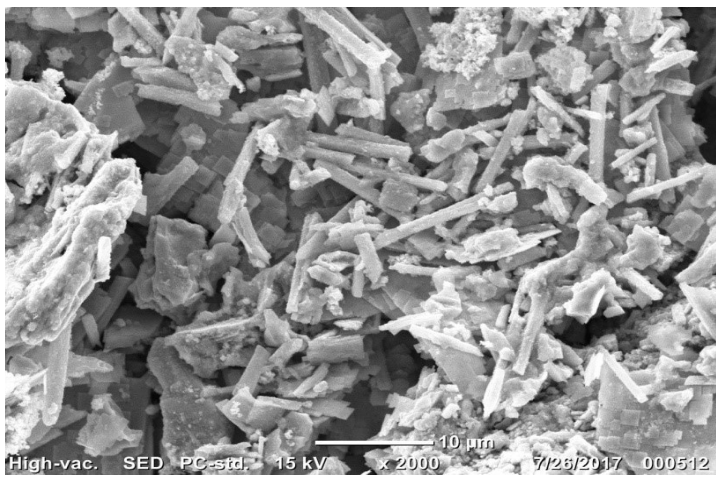

Figure 5. SEM image of SnO_2 nanorod annealing at 600ºC

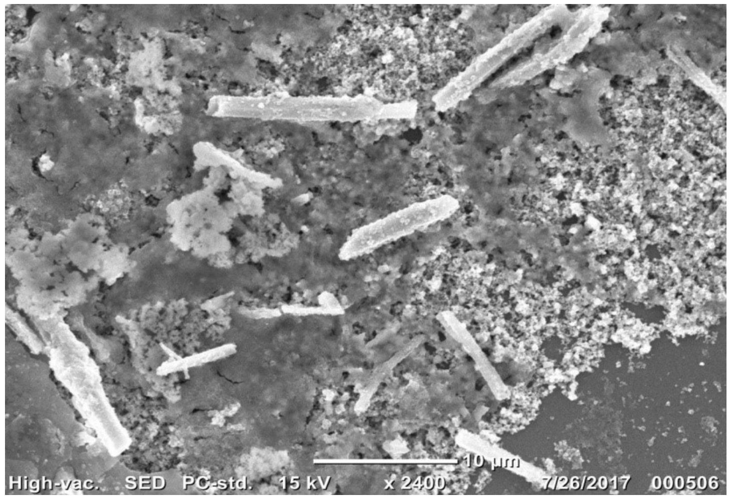

Figure 6. SEM image of SnO_2 nanorod annealing at 550ºC

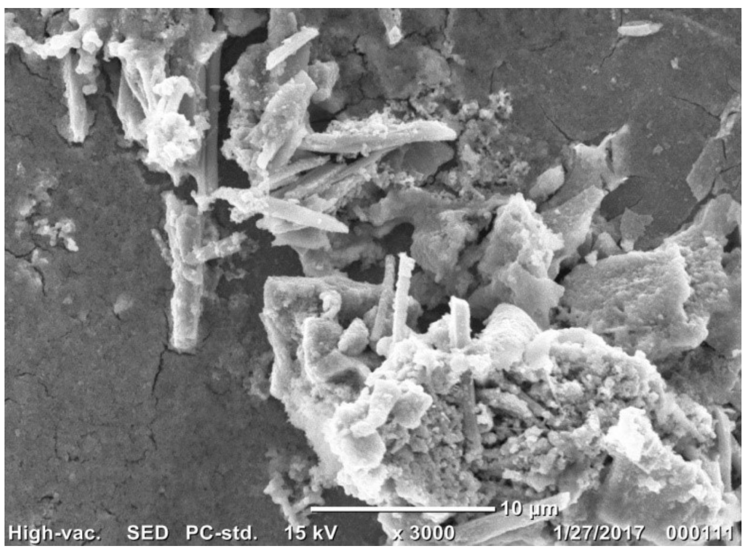

Figure 7 . SEM image of SnO_2 nanorod annealing at 500ºC

Application of SnO_2 nanorod

Preparation of natural dye

The natural dye extracted with methanol by the following procedure; Fresh leaves of tamarind (figure 8) were washed with water and dried (figure 9) at room temperature. Then, they were crushed into powder. Each powder (0.8g) was dissolved in the beaker and 25ml of methanol was added. The solution was annealed at 80°C for 1h by using water bath figure10. And then, the residual (solid) parts were filtered out and the resulting filtrates were used as dye solutions.

Next, optical properties of dye in UV and visible regions were analyzed by using Shimadzu UV-170 spectroscopy.

Figure 8.Tamarind dried leaves and powder

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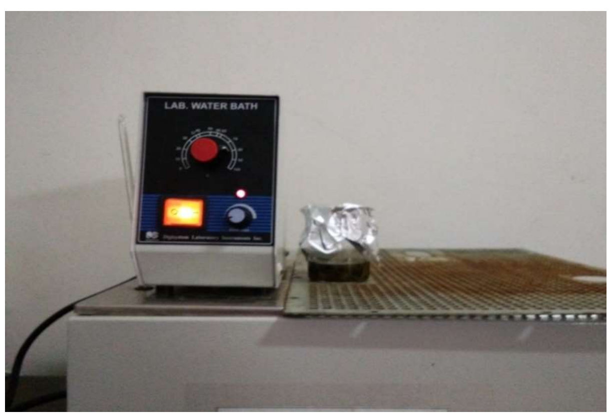

Figure 10. Annealing dye solution on water bath at 80 °C

UV – Vis Spectroscopic Study

The UV-Vis photospectra of tamarind dye were recorded with respect to the bare substrate placed in the reference beam using beam spectrophotometer in the range 400 to 700 nm.

Figure 11 show the UV visible absorption spectra of tamarind leaves dye at 80 °C. In the figures, chlorophyll dye has absorption features in UV light zone. In the visible light region zone, the absorption peaks of chlorophyll dye extracted from tamarind leaves at 80 °C lie at 674.40 nm. The energy band gap of wavelength was calculated by using Plank's photoelectric equation.

$$\mathbf{E} = \frac{h\nu}{\lambda} = \frac{hc}{\lambda}$$

where E = energy band gap (eV)

h = Plank's constant =6.625 x 10^{-34} J-s

c = 2.99 x 10^{8} ms^{-1}

λ = the wavelength (nm)

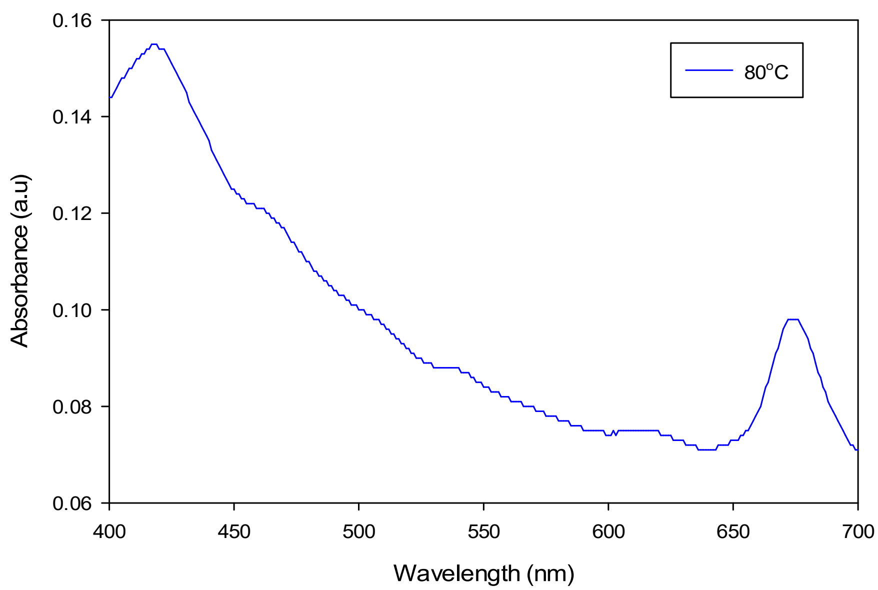

Figure 11. Absorbance spectra of natural dye extracted from tamarind leaves at 80ºC

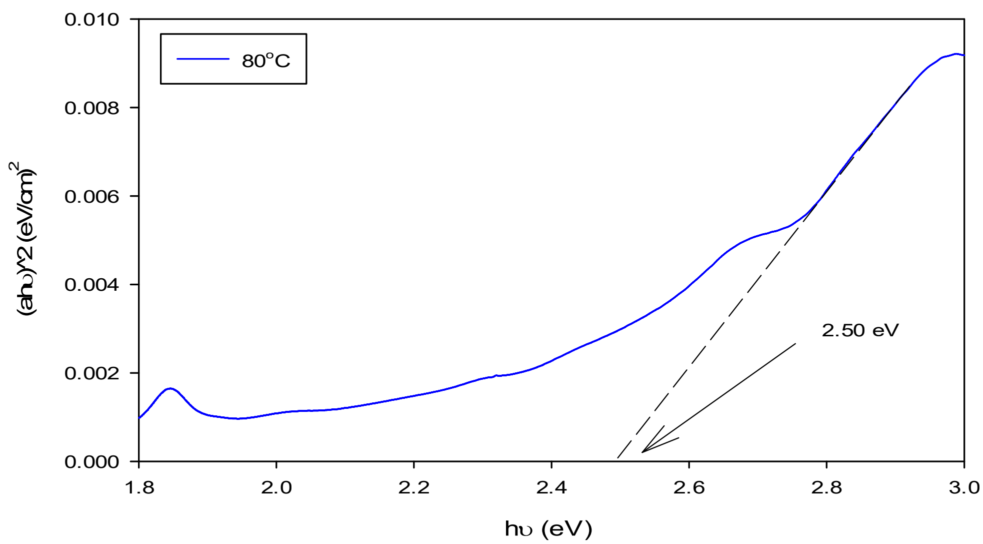

Figure 12. Absorbance spectra of natural dye extracted from tamarind leaves at 80 ºC

The transmission spectra were analyzed by plotting $(\alpha h\upsilon)^2$ Vs hυ , based on following equation.

$$\alpha h\upsilon = A(h\upsilon - E_g)^{n/2}$$

Where α is the absorption coefficient A is a constant (independent from υ) and n is the exponent that depends upon the quantum selection rules for the particular material. A straight lines (fig 12) were obtained when $(\alpha h\upsilon)^2$ is plotted against photon energy (hυ), which indicate that the absorption edge is due to a direct allowed transition (n=1 for direct allowed transition). The intercept of the straight line on hυ axis corresponds to the optical band gap (E_g) and its value was determined.

Fabrication of DSSC

For photoelectrode, SnO_2 nanorod onto FTO glass were immersed into the dye solution for 12h and then took off. For counter electrode, carbon paste was coated onto FTO substrate and annealed at 120 ºC for 15 min.SnO_2 nanorod photoanode and carbon counter electrode were sandwiched and two binder clips were used to hold the electrodes together. Iodine was added and it was used as a mediator. Alternately open and close each side of solar cell to draw electrolyte solution in and wet SnO_2 nanorod. Remove excess electrolyte from exposed areas. Fasten alligator clips to exposed sides of solar cell. Photocurrent – Voltage (I-V) curve and photovoltaic properties were investigated.

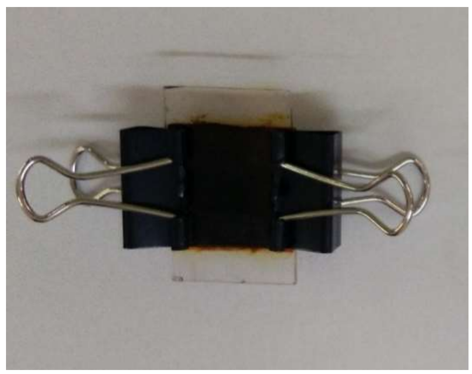

Figure13. Dye sensitized solar cell

Photovoltaic Properties

Figure 14 showed the J-V curves of SnO_2 nanorod for DSSCs with tamarind leaf powder dye solution. From the figure, the maximum power point was obtained by tangential point on J-V curve. By drawing the maximum power point onto X- axis , the maximum voltage (V_m) was obtained. By drawing the maximum power point onto Y- axis , the maximum current (I_m) was obtained. From the analysis, short circuit current (I_{sc}) , maximum current density (J_m), open circuit voltage (V_{oc}), maximum voltage (V_m) , conversion efficiency (η) and fill factor (FF) of dye sensitized solar cell was shown in Table 1.

Table 1. Photovoltaic properties of SnO_2 nanorod DSSC

Substrates	J_{sc} (mA/cm2)	V_{oc} (V)	*FF*	*PCE* (%)
FTO	15.89	0.13870	0.49	1.09

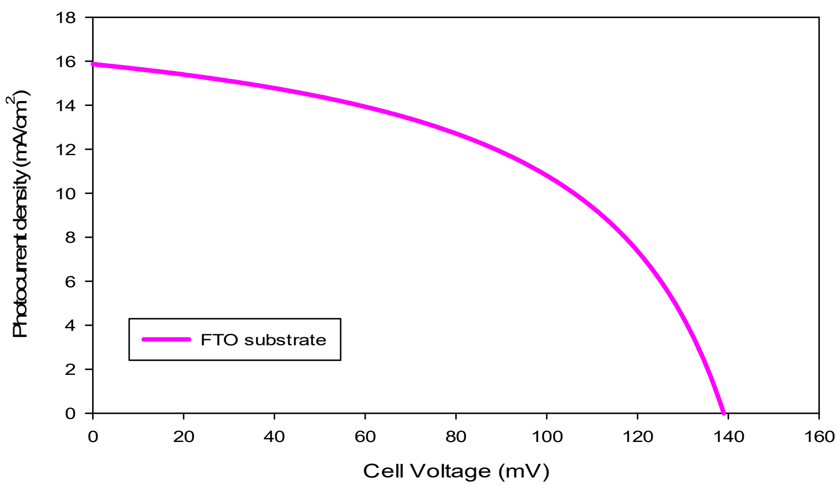

Figure14 . J-V curve of SnO2 nanorod DSSC (FTO glass)

Conclusion

SnO_2 nanorod were obtained by immersing of SnO_2 seed layer coated onto FTO glass substrate into the aqueous solution by using chemical bath deposition method. According to the XRD result, SnO_2 powder were successfully formed with tetragonal symmetry. After calcination at 500°C, the SnO_2 nanorod showed one – dimensional structure with the diameter of 300 nm –700 nm and length reaches up to around 5µm. At 550°C and 600°C , the diameters and lengths were nearly the same as the SnO_2 nanorod at 500°C. In 550°C, very few of SnO_2 nanorod were appeared. In 600°C , a lot of SnO_2 nanorod were appeared and more smooth than the product at 500°C. In the SEM image, the products with a closer view comprises straight structures at different temperature. So, SnO_2 nanorod at 600°C is better than others. When the SnO_2 nanorod for chlorophyll – based natural dye sensitized solar cell was fabricated, power conversion efficiency (1.09 %) was obtained.

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