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Dye-Sensitized Solar Cell using Extract of Jacaranda Mimosifolia and Salvia Spelendens as a Natural Sensitizer

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Abstract

Nanocomposites of Sn-Ti oxide were prepared by sol-gel method to use as photo anode for dye sensitized solar cell (DSSC). Crystal size, elemental contents, structural properties and energy band gaps of the as-synthesized powders was examined using XRD, EDX, SEM and UV-Vis spectrophotometer respectively. A quasi-solid state photo electrochemical solar energy conversion device with I_3 /I redox couple has been constructed and characterized. Natural pigments were also extracted using ethanol, HCl and water solvents from *jacaranda mimosifolia* and *Salvia spelendens* to use as sensitizer. Ethanol extract of natural sensitizer absorbs in the visible region. Dye sensitized solar cells were assembled using extracted natural dyes. 8.63% Incident photon to current conversion efficiency (IPCE) at 330 nm was obtained. The photo electrochemical performance of the quasi-solid state DSSCs based on the ethanol extract of *Salvia spelendens* showed best photon conversion efficiency with an open circuit voltage (V_{oc} = 266 mV) and short circuit current density (J_{sc} = 0.1930 $mAcm^{-2}$) at 100 $mWcm^{-2}$ white light intensity.

Keywords: Sol-gel method, photo electrochemical cell, dye sensitized solar cell, quasi -solid state electrolyte

1. Introduction

Almost 90% of the world energy is sourced from fossil fuels which mainly fall into oil, natural gas and coal, however, the production of oil will soon not be able to keep up with the growing demand leading to dire economic consequences (Zhang *et al*., 2009). In addition, the combustion of fossil fuels has been implicated in anthropogenic global warming, which is predicted to produce widespread environmental damage. Development of renewable and clean energies sourced from solar, wind, geothermal, nuclear and tide should be of strategic importance (Gratzel, 2001). Light from the sun is arguably the ideal source of energy. The solar flux striking the earth contains 10000 times the average global power usage, and is the largest single source of clean energy which is readily available. While technologies have been developed to harness solar energy efficiently, they are not yet an economically viable alternative to fossil fuels (O'Regan and Grätzel, 1991).

Solar cells are one type of photovoltaic cells which use a method of generating electrical power by converting energy of light into direct current electricity using semiconductors that exhibit the photovoltaic effect (Park, 2010). Photovoltaic devices convert solar irradiation directly to electricity with zero emission and it has the potential to supply the whole world's energy demand (Grätzel, 2001). Many photovoltaic devices have already been developed over the past five decades (Goetzberger and Hebling, 2000; Goetzberger *et al*., 2003). However, wide-spread use is still limited by two significant challenges, namely conversion efficiency and cost (Bagnall and Boreland, 2008).

Silicon (Si) based solar cell devices have dominated the market for the past decades. However, the high demanding and processing cost of crystalline silicon have limited their applications and at the same time encouraged the development of simple and low cost alternatives. Dye-sensitized solar cells (DSSCs) utilizing nanostructured materials have been considered as one of the promising candidates for photovoltaic devices (O'Reganand Grätzel, 1991). The DSSCs are a photoelectrochemical system, which incorporate a porous-structured oxide film with adsorbed dye molecules as the photosensitized anode. A platinized fluorine-doped tin oxide (FTO) glass acts as the counter electrode (cathode), and a liquid electrolyte that traditionally contains I^-/I_3^- redox couples serves as a conductor to electrically connect the two electrodes (Gratzel, 2001; Kroon *et al* ; Longo, 2007).

Dye-sensitized solar cells based on nanoporous films of metal oxides have gained much attention as alternative approach to the silicon solar cells because of their prospect for the low cost photovoltaic energy conversion (O'Regan and Grätzel, 1991). In this contest, promising solar energy into electrical energy conversion efficiencies of more than 10% have been achieved for DSSCs based on TiO_2 (Nazeeruddin *et al*., 2001). However, the efficiency of DSSCs constructed with other candidates of metal oxides such as ZnO, SnO_2 etc., lies much more behind (Keis *et al.,* 2002).

Dye-sensitized solar cells have extensively employed TiO_2 semiconductor for efficiency development. Towards next evolution, however, design of new semiconductor material is the key for developing higher performance of DSSCs. Tin oxide with a large band gap (Eg =3.6 eV at 300 K) used as an important n- type semiconductor is quite attractive because of its excellent optical and electrical properties and chemical stability (Wang *et al.,* 2006). The DSSC of SnO_2 was reported with an efficiency of 1.13% with a shortcircuit current (J_{SC}) of 8.05 mA/cm^2 and a V_{oc} of 0.50 V (Tiwana *et al*., 2011). But the conduction band edge of SnO_2 locates at a lower level in the energy scale. Due to this reason, open circuit photovoltage of DSSCs constructed with SnO_2 are low compared to DSSCs constructed from TiO_2 or ZnO. (Tennakone *et al*., 1999 and Prasittichai *et al.,* 2010). Therefore, this study is to evaluate and compare the cell performance of SnO_2 semiconductor using a binary system SnO_2/TiO_2 nanocomposite and natural dyes extracted from *Salvia spelendens,* and *jacaranda mimosifolia*.

2. Experimental

2.1. Sol-Gel synthesis of $SnO_2/TiO_2/FeO_3$ Nanocomposite.

SnO_2/TiO_2 nanocomposite was synthesized by sol-gel method (Rohana *et al*., 2010) using precursors, tin tetrachloride penta hydrate ($SnCl_4.5H_2O$) and titanium tetra chloride ($TiCl_4$) using tin as host. Ammonium hydroxide, NH_4OH, was used for precipitation and ethyl alcohol was employed to wash the precipitate. 0.1M nanopowder was prepared by dissolving appropriate amount of precursor salts in 50ml water. 0.1M aqueous NH_4OH solution was added to the above solution by drop wise under constant stirring. After stirring for 2hrs the sol was aged at room temperature for 24hrs. The resulting gel was then washed with ethanol until the pH of solution become 7. The gel was dried at 80ºC for 24 hrs in order to remove water molecules. Then the samples were calcined at 400°C for 4hrs in order to reduce thermal shock. Under the same experimental conditions, various molar proportions (80%, 70% and 60% Sn mixed with 20%, 30%, and 40% Ti respectively) of the hydrous tin oxides, and titanium oxides powders were obtained.

2.2. Extraction of natural pigment

Fresh flowers of *Salvia spelendens* and *Jacaranda mimosifilia* were collected. Before drying, the leaves were washed with distilled water.The collected flowers were dried at room temperature in a shade to prevent pigment degradation. After drying for about 2 months the samples were crushed with Micro Plant Grinding machine to produce the powder of the respective plant materials.

Two gram of each powder sample was taken and soaked in 50 mL of ethanol for the ethanol extract. The same amount of powder was soaked in 50 mL water and HCl in separate bottle respectively. The solution was stored at room temperature for about 24 hrs to dissolve the powder completely. Then the solution was filtered with glass filter to separate the solid from the pure liquid. Optical absorption of each extract was measured by taking small amounts of the filtered samples.

2.3. Characterization of SnO_2/TiO_2 nanocomposite

Crystal size, elemental contents, structural properties and energy band gaps of the as-synthesized powders was examined using XRD, EDX, SEM and UV-Vis spectrophotometer respectively. The absorption characteristics of natural dye extracted from *Salvia spelendens* and *Jacaranda mimosifilia* were also characterized by UV.Vis spectrophotometry.

2.4. Solar cells assembly

ITO-coated glass was employed as a substrate for the photoactive materials and counter electrodes. It was cleaned in ultrasonic bath successively with acetone (Aldrich), 2-propanol (Riedel-de Haen), and ethanol (BDH). 3 g of SnO_2/TiO_2 powder was ground in a porcelain mortar. After grinding 4-5 ml of the PEG solution in Acetonitrile was slowly added to the powder and completely mixed by using the mortar. Finally, a detergent 0.05 mL Triton X-100 (Aldrich), was added to facilitate the spreading of the colloid on the substrate. The ITO was covered on two parallel edges with adhesive tape to control the thickness of the paste film and to provide noncoated areas for electrical contact. The colloid was applied to one of the free edges of the conducting glass and distributed with a glass rod sliding over the tape-covered edges. After air drying, the electrode was sintered for 30 min at 450°C in a furnace. Coating of the SnO_2/TiO_2 surface with dye was carried out by soaking the film for 6 hrs in ethanol extracted natural pigments. The dye coating was done immediately after the high temperature annealing while still hot in order to avoid rehydration of the SnO_2/TiO_2 surface. After completion of the dye adsorption the electrode was withdrawn from the solution. It was stored in dry ethanol or immediately wetted with redox electrolyte solution for testing.

The polymer film for the counter electrode was formed by electrochemical polymerization of 3,4-ethylenedioxythiophene (EDOT) (Aldrich), in a three electrode one-compartment electrochemical cell. The electrochemical cell consisted of a pre cleaned ITO-coated glass working electrode, platinum foil counter electrode and quasi- Ag/AgCl reference electrode. The solution used for the polymerization contained 0.2 M EDOT and 0.1 M $(C_2H_5)_4NBF_4$ (Aldrich) in acetonitrile (Aldrich). The polymerization was carried out potentiostatically at +1.8 V for 2 seconds. At this potential, the electrode surface becomes covered with blue doped PEDOT film. The PEDOT covered ITO glass was then rinsed with acetonitrile and dried using hair dryer.

The polymer gel electrolyte was prepared as follows (Kang *et al.*, 2010): 0.9 M of 1-ethyl-3-methyl immidazolium iodide was added into acetonitrile under stirring to form a homogeneous liquid electrolyte. In order to obtain a better conductivity, 0.5 M of sodium iodide was dissolved in the above homogeneous liquid electrolyte, and then 0.12 M iodine and 35% (w/w) of PVP were added. Then, the resulting mixture was heated at 70 – 80^{o}C under vigorous stirring to dissolve the PVP polymer, followed by cooling down to room temperature to form a gel electrolyte.

Finally, the polymer electrolyte complexed with I_3^-/I^- was deposited in the form of thin film by solvent casting on top of the dye coated $SnO_2/TiO_2/Fe_2O_3$ electrode and allowed to dry. The PEC was completed by pressing against PEDOT-coated ITO glass counter electrode. The PEC was then mounted in a sample holder inside a metal box with 1 cm^2 opening to allow light from the source. All experiments were carried out at room temperature. The photoelectrochemical measurements of the cell were performed using a computer controlled CHI630A Electrochemical Analyzer. A 250-W tungsten–halogen lamp regulated by an Oriel power supply (Model 68830) was used to illuminate the PEC.

3. Result and Discussion

3.1. X-ray diffraction analysis

X-ray diffraction is a convenient method for determining the mean size of nano crystallites in nano crystalline bulk materials. Four series of as-synthesized powders were examined by XRD.The diffiraction peaks of all the powders are shown in Figures 1. The results show distinct peaks with their corresponding 2θ and β values (given in units of degree and radians, respectively) which accounts for the crystalline nature of all the as-synthesized powders. All diffraction peaks can be readily indexed to tetragonal SnO_2 nanoparticles. The diffraction peaks are markedly not intense,which is due to low calcination temprature. The average crystalline size was estimated using Dubye Scherere equation.

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad \textbf{(1)}$$

Where: *D* is crystallite size in nm, *K* is the shape factor constant usually 0.89, β is the full width at half maximum(FWHM) in radians of 2θ, λ is the wave length of the X-ray which is 0.15406 nm for Cu target $K\alpha_1$ radiation and θ is the Bragg's angle.

Using Equation (1), the average crystallite sizes of the as synthesized semiconductors were calculated and given in Table 3.1.1.

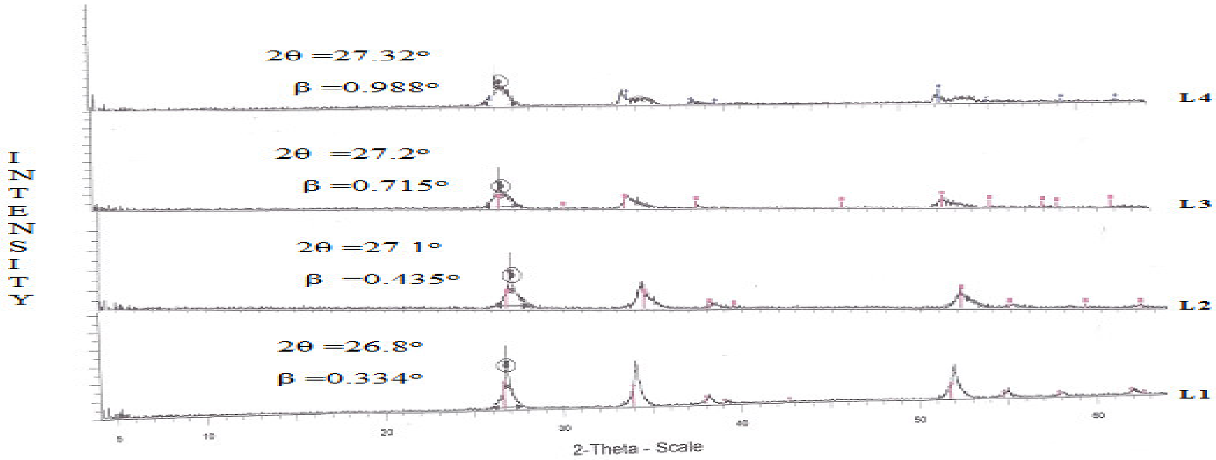

Figure1. XRD pattern of as-synthesized powders of Sn-Ti at different ratio.

Table 1. Crystal size of as-synthesized powders

Nanocomposite	2θ degree	β (radians)	D (nm)
L1	26.8	0.0057934	24.5
L2	27.1	0.00759075	18.73
L3	27.2	0.01247675	11.4
L4	27.32	0.0172406	8.25

From Table 1 all the as-synthesized powders fall in nano range and all are crystalline. L1 for pure oxide, L4 have got the smallest crystalline size 24.5 and 8.25 nm respectively. Hence, the smallest the crystal size, the greatest the surface area of the powders the lager surface area may benefit the contact between dye stuff and light, and can absorb larger numbers of dye stuff as well as increase photo activities. The smaller particle size for the mixed oxide could be attributed to an increase in the thermal stability and the resistance to sintering caused by the doped oxide. Based on this fact L1 and L3 are selected and subjected for further study.

3.2. Optical absorption measurements of the as-synthesized samples

The absorption edges of the as-prepared powders were determined using UV-Vis spectrophotometer. UV-Vis absorption spectra for the selected synthesized powders are shown in Figure 2 below. Band gap energy (E_g) of the as-synthesized powders was determined by the following equation.

$$E_g = \frac{1240\ eV}{\lambda_{max}} \qquad (2)$$

Where, E_g is bandgap energy in electron volts and λ_{max} is wavelength (nm) corresponding to absorption edge.

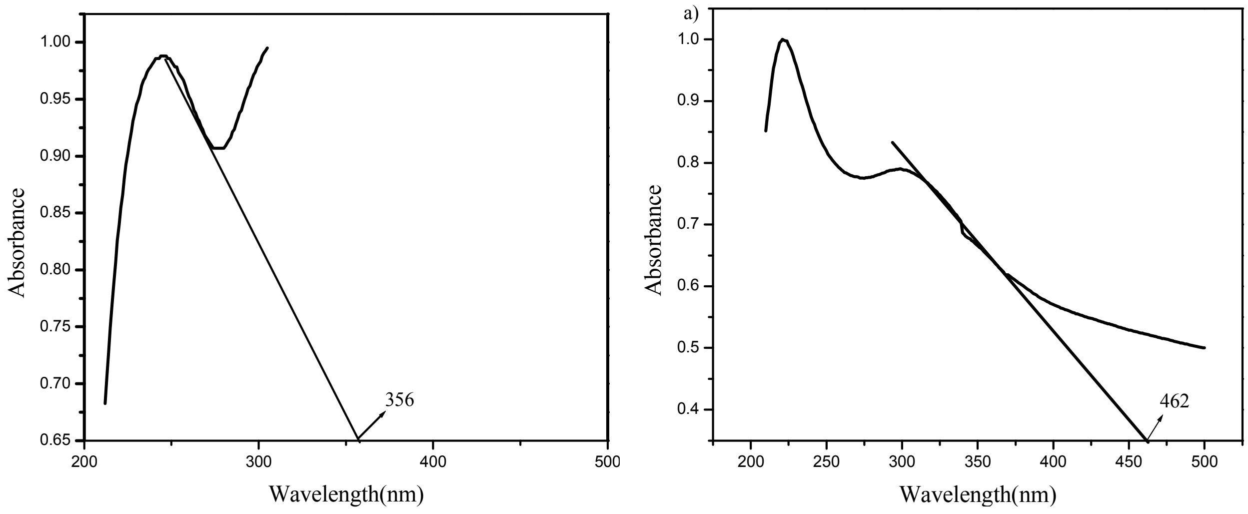

Figure 2. UV-Vis absorption spectra of as-synthesized nanocomposite a) L1 and b) L4

From the above graphs it is evident that, the absorption edges of the binary oxide (L4) shift remarkably to the visible range relative to the pure Oxide (L1). The energy band gap of the as-synthesized nanocomposite is 3.48 and 2.68 for L1 and L4 nanocomposite respectively.

3.3. Elemental analysis of the powders

The major components of the as-synthesized powders were confirmed using energy dispersive X-ray spectroscopy (EDX). The EDX spectrum L1 and L4 nanocomposite was given in figure 3 and 4 below respectively.

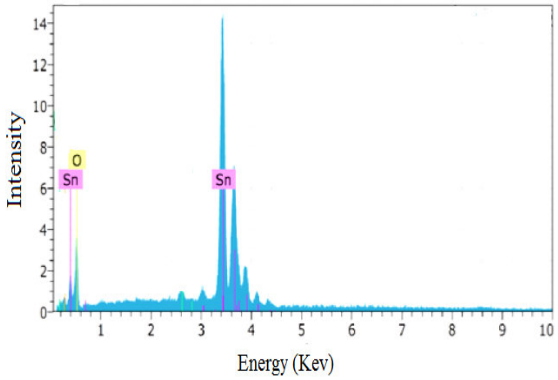

Figure-3 EDX Spectrum of L1

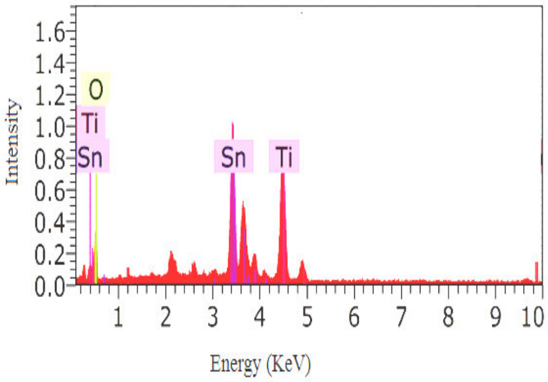

Figure-4 EDX Spectrum of L4

It is depicted from the above spectrum that the powders named as L1 is enriched with only oxides of Tin while L4 is enriched with oxides of Sn-Ti. Elemental content was quantitatively analyzed through EDS. The atomic percentage of the as-synthesized powders is summarized in the Table 2 below.

Table-2. .Atomic percentage of each element of the as-synthesized powders

Sample	Atomic %		
Name	O%	Sn%	Ti%
L1	66.74	33.26	-
L4	67.12	16.59	16.29

3.4. SEM analysis of the as-synthesized powders

Scanning electron microscopy(SEM) was used to observe the morphological features of the selected nanopowders. SEM images of SnO_2 and SnO_2/TiO_2 are presented in Figure5 below.

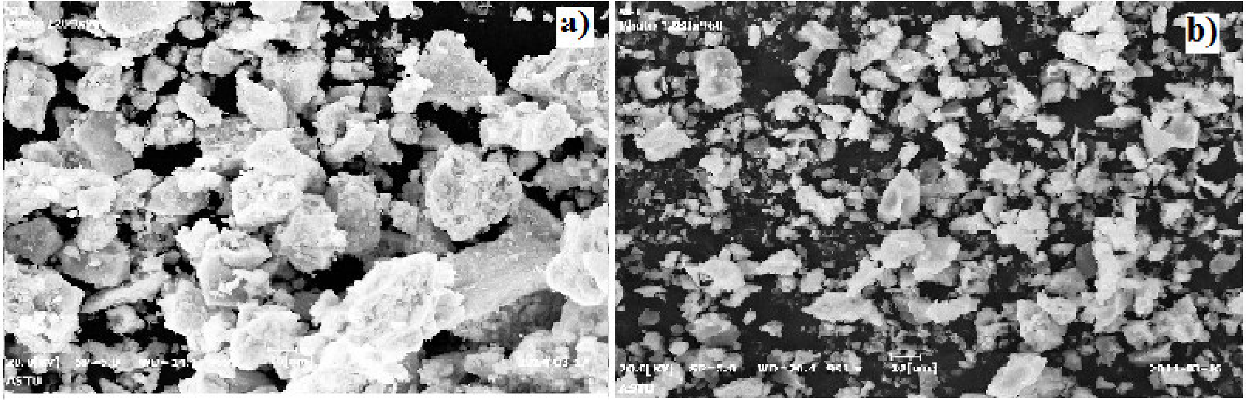

Figure-5. Scanning electron microscopy images of a) L1 b) L4

It is evident that, from the SEM analysis the fabricated nanocomposite have different morphology. SnO_2(a) particles are of irregular shape and non uniform size. The particles have highly mesoporous foam like structure and agglomerated to nearly spherical morphology. SEM of binary oxide composite(b) displays white snow-like flakes of SnO_2 mixed with TiO_2.

3.5. Optical Absorption Measurements of the Natural Dyes

The absorption edges of the prepared dyes were measured by using UV-Vis spectrophotometer. The graph of absorbance versus wavelength of each sample is shown in Figures 6&7 below. Both ethanol extract of *Jacaranda mimosifolia* and S*alvia spelendens* shows an absorption peak at 665 nm and 666 nm respectively this absorption peaks related to the characteristics absorption peak of chlorophyll b which shows an absorption peak in between 600 - 700 nm. All water extracts of both the plant material doesn't show any absorption peak, this does not mean that there is no associated pigment. Instead it is because, the anthocyanin which can exist in two form, one in flavylium cation and the other quinonoidal when the quinonoidal forms are dominant the pigment did not show absorption maxima.

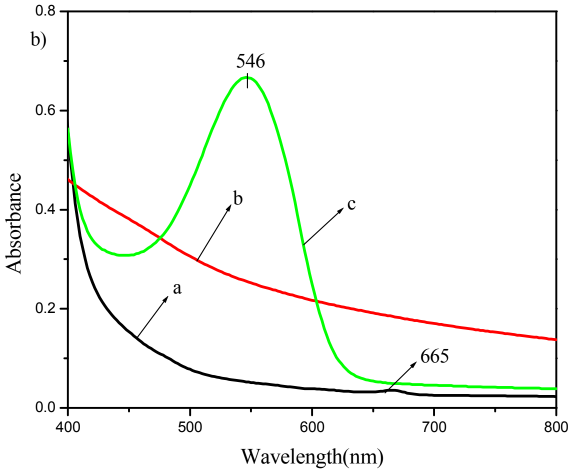

Figure-6 Absorption spectra of *Jacaranda mimosofilia* extracted with a) ethanol b) water c) HCl

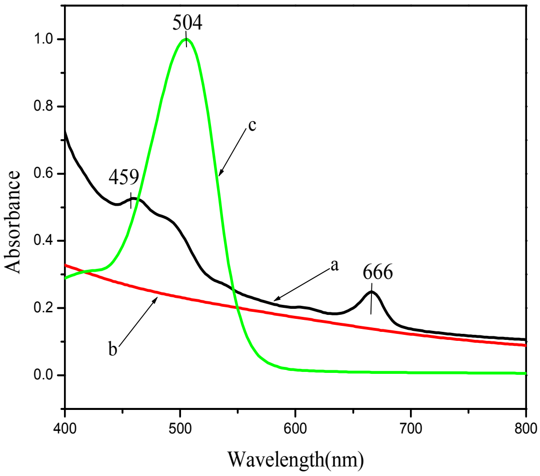

Figure-7 Absorbtion spectra of *Salvia spelendens* extracted with a) ethanol b) water c) HCl

Based on the above reason, absorption spectra measurements of 0.1 M HCl extracts of all the natural dyes were also performed (figure 6 and 7) Hence, all flowers shows absorption peak at different wavelength.

Even though all flowers show an absorption peak in visible region with acid extract, it is known that SnO_2 nanoparticles easily dissolves in acidic medium and affects the device construction. Therefore, only ethanol extracts of all the natural dyes are selected for the solar energy conversion.

3.6. Current density versus voltage characteristics

Photovoltaic tests of DSSCs using these natural dyes as sensitizers were performed by measuring the current density–voltage (J – V) curves under irradiation with white light (100 mWcm^{-2}). The performance of natural dyes as sensitizers in DSSCs was evaluated by short circuit current density (J_{sc}), open circuit voltage (V_{oc}), fill factor (FF), and energy conversion efficiency (η). The typical current density - voltage (J – V) curves of the DSSCs using the sensitizers extracted from flowers of *jacaranda mimosifolia* and *Salvia spelendens* are shown in Figure 8 and 9 respectively.

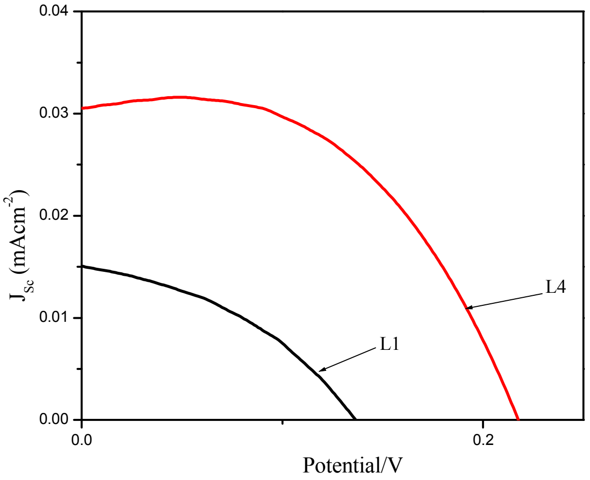

Figure-8. The J-V characteristics of ethanol extracted *Jacaranda mimosifolia* sensitizer with L1and L4

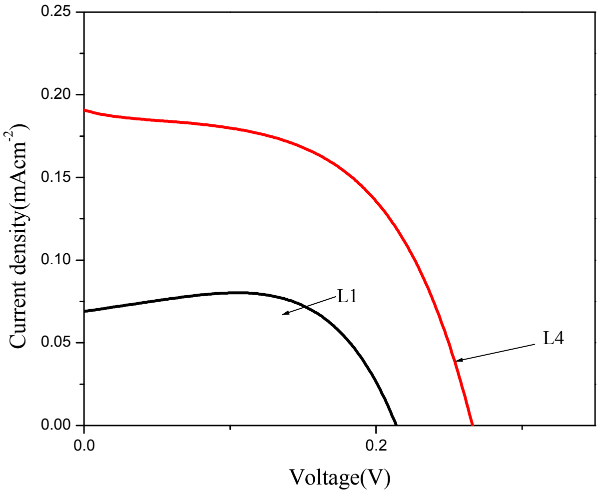

Figure-9. The J-V characteristics of ethanol extracted *Salvia Spelendens* sensitizer with L1and L4

Table-3.. Photovoltaic performance of L1and L9 based DSSCs with natural pigment sensitizers

Sensitizer	Semiconductor	J_{sc} (mA.cm^{-2})	V_{oc} (V)	FF %	η
Jacaranda mimosifolia	L1	0.01499	0.136	40.0	0.00081
	L4	0.03411	0.221	52.6	0.00397
Salvia	L1	0.07100	0.214	56	0.015
spelendens	L4	0.19300	0.266	77	0.0513

An open circuit voltage (V_{oc} = 266 mV) and short circuit photocurrent density (J_{sc} = 0.193 mAcm^{-2}) were obtained for the DSSC sensitized by the ethanol extract of *Salvia Spelendens*; the efficiency of this DSSC reached 0.0513.

3.7. IPCE Characteristics

The incident monochromatic photon to current conversion efficiency (IPCE), defined as the number of electrons generated by light in the external circuit divided by the number of incident photons at each wavelength. The IPCE of DSSCs with each semiconductor composite was determined using Equation (3).

$$\text{IPCE\%}=\frac{1240[\text{eVnm}]\text{譐}_{\text{SC}}[\text{mAcm}^{-2}]}{\lambda[\text{nm}]\text{譏}_{in}[\text{mWcm}^{-2}]}?\ 00 \qquad \textbf{(3)}$$

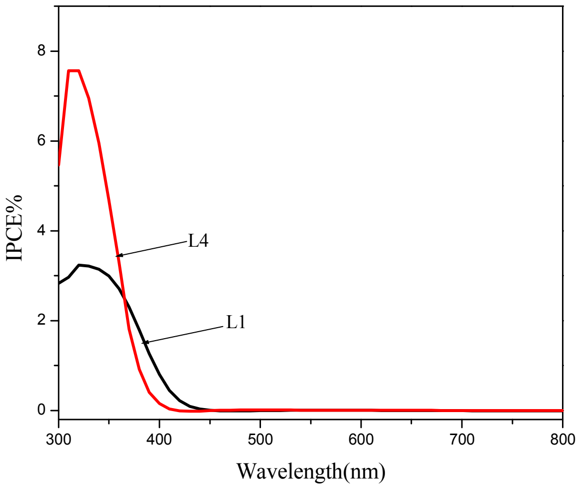

Figure-10. IPCE spectra of *Salvia Spelendens flower* based on L1and L4

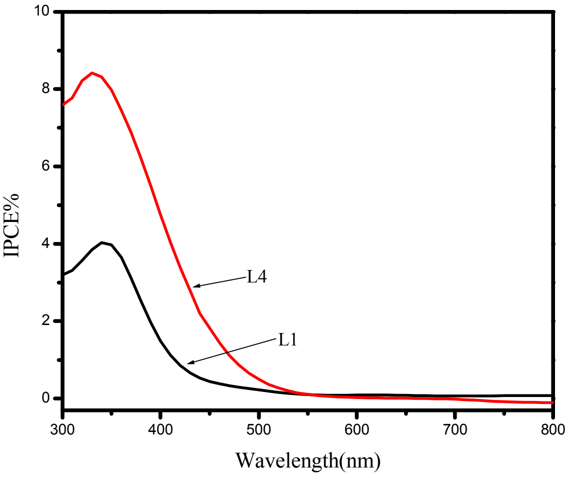

Figure-11. IPCE spectra of *Jacaranda mimosofilia flower* based on L1 and L4

From the photoaction spectra above, maximum IPCE percentage obtained for the natural dyes was 8.63% at 330 nm was obtained for the device based on SnO_2/TiO_2 semiconductor. Natural dyes suffer from low V_{oc}, J_{sc} which leads lower power conversion efficiency than an equivalent commercial dye sensitized cell. This is because of most of natural dyes didn't completely attach to the SnO_2 nanopartciles, even upon washing by solvent to avoid aggregation from the socked film, the sensitizers leave the surface of the film, this is may be due to, more of the natural dye attached with film of the nanoparticles by physical attaching or by a weak bonding force.

4. Conclusion

In this work, SnO_2/TiO_2 semiconductor nanocomposite was successfully synthesized and characterized. The photoelectrochemical performance of the quasi-solid state DSSCs based on the ethanol extract of *Salvia spelendens* showed best photon conversion efficiency with a V_{oc} of 266mV,J_{sc} of 0.1930 $mAcm^{-2}$ at light intensity of 100 $mWcm^{-2}$. With ethanol extract of *salvia spelendens* and *jacaranda mimosifolia* with L4 nanocomposite 0.0513 and 0.00397 efficiencies was obtained respectively. This study indicates that it is possible to use binary metal oxide semiconductors for photon conversion as they witnessed better performance compered to single oxide systems.

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