

Effect of Ligands attached to Fluorescein on the Photocurrent of Solid-State Dye-Sensitized Solar Cells

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Abstract: *Organic dyes have received much attention due to environmental compatibility. Fluorescein is one of the environmentally friendly dyes. The effect of ligands attached to fluorescein on the photo-performance of TiO₂/dye/p-semiconductor type solar cells was examined.*

Keywords: organic dyes, power conversion efficiency, solid-state cells, effect of the ligands

1. Introduction

Energy crisis has become a threatening issue in the current world and many alternative power sources have been identified. Solar-energy is one of promising green energy from those choices. Solar cell is a simple device that converts solar energy in to electricity. The first-generation solar cell based on silicon and initiated in nineteen-fifties [1]. However, due to complicated manufacturing process of this type of solar cells, scientists explored different types of solar cells. As a result, a thin film consisting of amorphous silicon, CuInS₂ or CuInSe₂ solar cells are identified as second-generation solar cells [2]. Dye sensitized solar cells became as the third generation of solar cells and firstly demonstrated by a Swiss research group in 1981 [3]. Since then, large numbers of dye-sensitized solar cells have been reported in this field. The dyes have been composed with different chromophores and also having different surface chelating groups for making bonds with metal oxide semiconductors. In this study, the effect of ligands attached to fluorescein was examined on the photocurrent generation of TiO₂/dye/p-semiconductor type solar cells. These dyes sensitized solid-state cells produce photocurrent of 100 μAcm^{-2} range to 4.0 mAcm^{-2} varying with the ligand attached to the fluorescein molecule. Among the dyes used in this work, mercurochrome produced comparable photocurrent generation in TiO₂/sensitizer/p-semiconductor type solar cells. The maximum photocurrent of 9.0 mAcm^{-2} was observed for the solid-state cell composed of mixture of dyes.

2. Materials and Methods

2.1. Deposition of compact TiO₂ layer on conducting glass plates

Fluorine doped tin oxide coated conducting (FTO) glass plates (Sigma–Aldrich) were cut into 1x2.5 cm² pieces, cleaned by detergent, thoroughly washed with distilled water and dried in an oven. One of the edges of FTO (1x1cm²) was covered with 3M-tape and placed horizontally on a hotplate facing the conducting side to air. Temperature of the hotplate was gradually increased to 450 °C. A solution of titanium diisopropoxide bis(acetylacetonate) 75wt% (Sigma-Aldrich) in isopropanol (1:9) was sprayed over the FTO quickly at the thermal equilibrium at 450°C and allowed to each to room temperature by disconnecting power of the hotplate.

2.2. Deposition of mesoporous TiO₂ layer on the compact layer

A 5.5 ml of glacial acetic acid and 5 ml of tetraisopropyl titanate (Sigma-Aldrich) were mixed with 10 ml of 2-propanol. 3 ml of distilled water was added to the above mixture followed by adding TiO₂ powder (0.65 g, Nihon Aerisol) and kept under vigorous stirring for 2-3 h. Resulted TiO₂ semi-colloidal suspension was used as the stock solution. A small amount of this stock solution was spread on compact TiO₂ layer coated FTO preheated at ~150°C by a plastic dropper and allowed to dry for few minutes. TiO₂ coated glass plate was fired at 550°C for 30 min and taken out after reaching them to room temperature by shutting down the power supply of the furnace. Loosely, bounded crust was removed by wiping TiO₂ film smoothly by piece of cotton wool. The thickness of TiO₂ film was achieved as 10 μm by repeating the coating procedure, successively.

Finally, TiO₂ coated conducting glass plates were cleaned by washing with acetonitrile.

2.3. Dye coating procedure on TiO₂ film

Dyes used in the present experiment were purchased from well-reputed manufactures and used as purchased. Dyes were dissolved in methanol (especially if not mentioned in the text) until concentration reaches $\sim 10^{-5}$ M. Dye was coated on TiO₂ electrodes as follows: TiO₂ coated glass plates were kept immersed in the dye solution and temperature of the dye solution was maintained as 40 °C. Dye coating process was carried out in an oil bath. Dye amount on TiO₂ electrodes was varied by controlling of the immersion time in the dye solution.

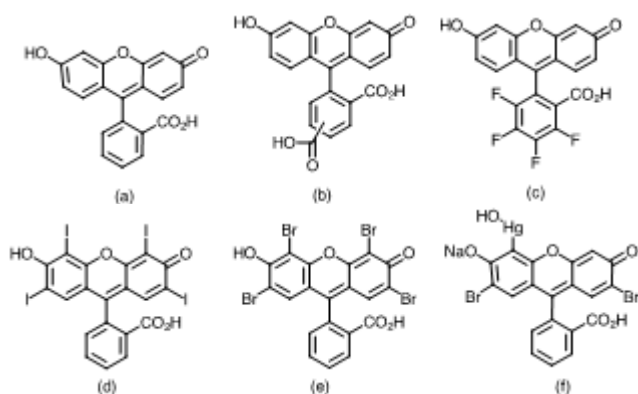
2.4. Coating procedure of hole-conductor on dyed TiO₂ film

TiO₂|dye|p-semiconductor cells were prepared by depositing CuI (Nacalai Tesque) on dye coated TiO₂ electrodes as the hole-conductor. CuI was mixed with 15 ml of moisture-free acetonitrile and excess CuI was allowed to precipitate. The filtrate was separated. A single drop of triethyl-amine-hydro-thiocyanate was added to the CuI solution as the surfactant. A small amount of above solution was carefully spread on the surface of the dye-coated TiO₂ plate heated at 150°C. This procedure was repeated until the conductivity of the CuI film reaches 50 Ωcm⁻¹. In some cases, CuCNS was used as the hole-conductor. Deposition of CuCNS on dye-coated films has been thoroughly discussed in elsewhere [4].

2.5. Measurements

Absorption spectra of dye solutions and dye coated TiO₂ films were measured by using UV-VIS-NIR spectrometer (Jasco V-570). The cell was constructed by pressing a Ni-coated FTO glass plate on the TiO₂|dye|CuI electrodes. Photo-effects of the cell were studied by illuminating the cell through TiO₂ layer. Current-voltage characteristics were recorded with a solar simulator (Wacom) coupled with semiconductor parameter analyzer (Hewlett Packard HP 4145B), under 1.5 AM condition. Scan speed was maintained as 0.01 Vmin⁻¹.

3. Results and Discussion



Scheme 1: Molecular structure of (a) fluorescein, (b) 5(6) carboxyfluorescein, (c) 4,5,6,7-tetrafluorofluorescein (d) erythrosin B, (e) eosin Y, (f) mercurochrome and (g) rose bengal

Molecular structure of used dyes (a) fluorescein, (b) 5(6) carboxyfluorescein, (c) 4,5,6,7-tetrafluorofluorescein (d) erythrosin B, (e) eosin Y, (f) mercurochrome and (g) rose bengal are shown in Scheme 1. We have divided these dyes

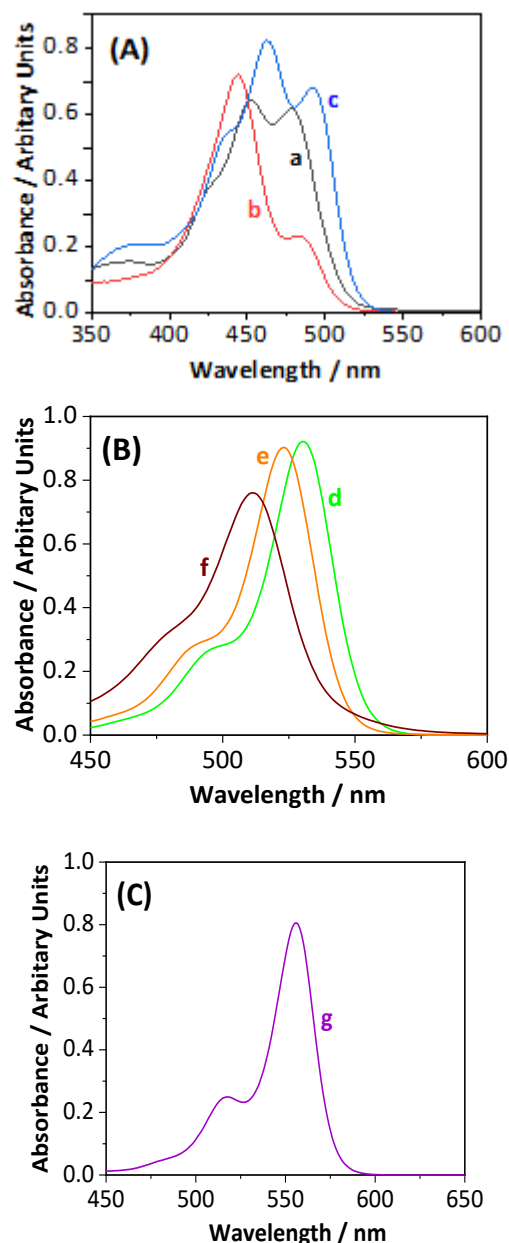


Figure 1: Absorption spectrum of (a) fluorescein, (b) 5(6) carboxyfluorescein, (c) 4,5,6,7-tetrafluorofluorescein (d) erythrosin B, (e) eosin Y, (f) mercurochrome and (g) rose Bengal, in methanol.

into three groups depending the position of ligand(s) attached. Absorption spectra of group-I dyes (a) fluorescein, (b) 5(6) carboxyfluorescein, (c) 4,5,6,7-tetrafluorofluorescein are shown in Fig. 1(A). Absorption spectra of group-II dyes (d) erythrosine B, (e) eosin Y, (f) mercurochrome are shown in Fig. 1(B) and group-III dyes (g) rose bengal is shown in Fig. 1(C). These dyes absorb visible light in different portion of wavelength regions. The onset of absorption spectrum of 5(6)-carboxyfluorescein has greatly shifted to the shorter wavelength region to compare to that of rose bengal. The onset of absorption spectra of other dyes is line up in the order of fluorescein, 4,5,6,7-tetrafluorofluorescein, mercurochrome, eosin Y, and erythrosine B. Solubility of these dyes was studied in different solvents. A higher degree of solubility of rose bengal, erythrosin B, eosin Y and mercurochrome was observed in all the solvents that were used in the present experiment. whereas, a poor degree of solubility of fluorescein, 5(6) carboxyfluorescein and 4,5,6,7-tetrafluorofluorescein was observed in acetonitrile.

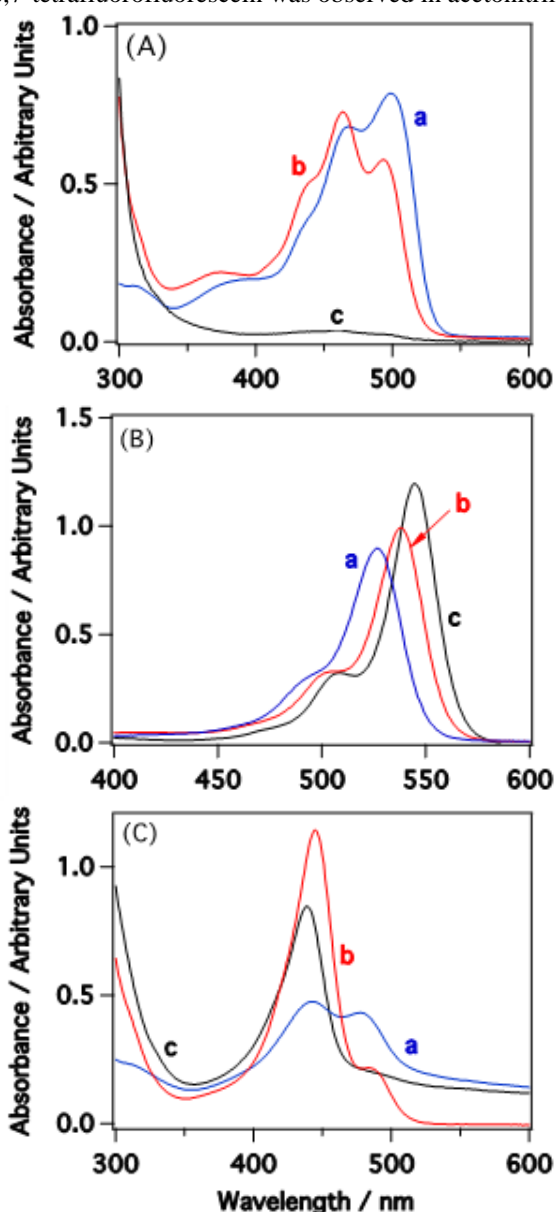


Figure 2: Absorption spectra of (A) 4,5,6,7-tetrafluorofluorescein, (B) erythrosin B and (C) 5(6) carboxyfluorescein in (a) water, (b) methanol and (c) acetonitrile

We have measured the absorption spectra of these dyes in different solvents. A blue shift in the maximum absorption (λ_{max}) was observed for 4,5,6,7-tetrafluorofluorescein in methanol (or acetonitrile) compare to that of the dye in water. Absorption spectra of 4,5,6,7-tetrafluorofluorescein in different solvents are shown Fig. 2(A). A red shift in the λ_{max} was observed for erythrosine B, eosin Y, mercurochrome and rose bengal in methanol (or acetonitrile) compare to that of the dyes in water. For example, absorption spectra of erythrosine B in different solvents are shown Fig. 2(B). However, such a significant shift was not observed for fluorescein and 5(6) carboxyfluorescein [Fig. 2(C)]. The shift in λ_{max} and different degree of solubility of these dyes due to difference of polarity of solvent may be associated with the Vander Val forces among dye molecules and the solvent [5,6].

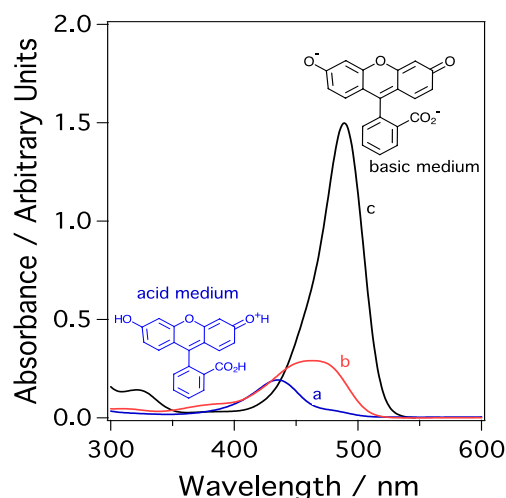


Figure 3: Absorption spectra of fluorescein, (a) acidic, (b) natural and (c) basic pH in water

We have studied the effect of pH with the absorption of these dyes. Except 5(6)-carboxyfluorescein, absorbance of other dyes has greatly reduced in acidic media. For an example, absorption spectra for fluorescein in different pH are shown in Fig. 3, (a). acidic, (b). natural and (c) basic media. A significant enhancement in absorption was observed in basic pH to compare to that of acidic and natural solutions. It is proposed that fluorescein di-anion at higher pH, mono-anion in moderate pH and dye mono-cation at lower pH [7]. The structures of dye di-anion and mono-cation are shown as an insert of Fig. 3. Absorbance of fluorescein, 5(6) carboxyfluorescein, 4,5,6,7-tetrafluorofluorescein and mercurochrome have greatly enhanced in basic media. However, absorbance of erythrosin B, eosin Y and rose Bengal remain unchanged in basic media. Luminescence properties of these dyes were also studied. Mercurochrome, eosin Y gave relatively higher luminescence properties, among the tested dyes, at room temperature. However, intensity of luminescence varies with the concentration of the dye solution. Luminescence spectrum of eosin Y is shown in Fig. 4, as an example. Generally, luminescence spectrum has red shifted to respect to the absorption spectrum. The luminescence spectrum of eosin Y shows mirror image of absorption spectrum and exhibited maximum Stokes shift of 26 nm for eosin Y. In the presence of TiO_2 in the medium a drastically reduction of luminescence was observed (not shown in the text),

indicating capability of injection of electron from excited dye molecules to TiO_2 particles.

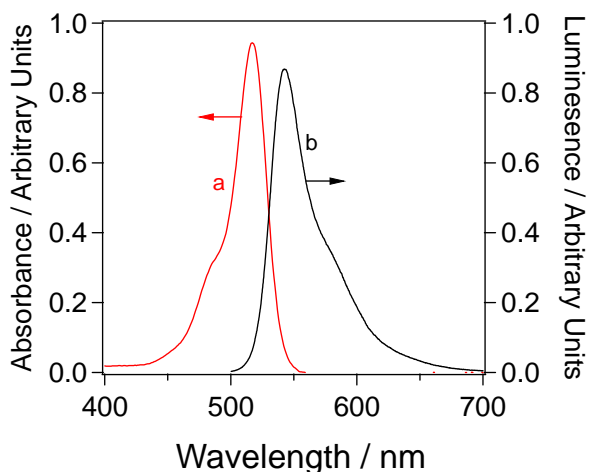


Figure 4: The (a) absorption and (b) luminescence spectrum of eosin Y in ethanol. Excitation wavelength was 450 nm.

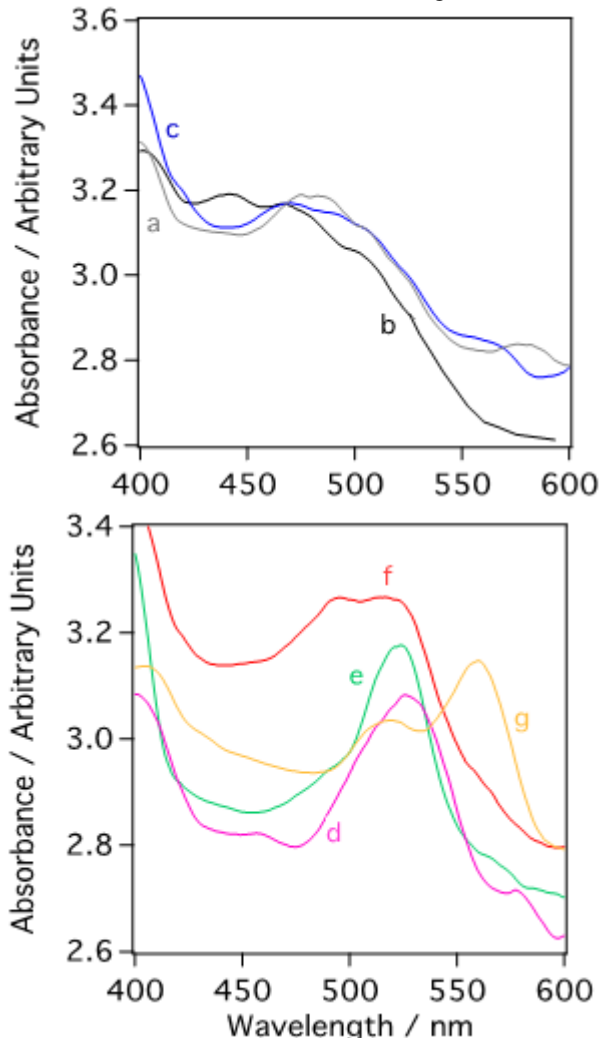


Figure 5: Absorption spectrum of (a) fluorescein, (b) 5(6) carboxyfluorescein, (c) 4,5,6,7-tetrafluorofluorescein (d) erythrosin B, (e) eosin Y, (f) mercurochrome, and (g) rose bengal coated TiO_2 electrodes

Absorption spectrum of (a) fluorescein, (b) 5(6) carboxyfluorescein, (c) 4,5,6,7-tetrafluorofluorescein (d) erythrosin B, (e) eosin Y, (f) mercurochrome and (g) rose bengal coated TiO_2 electrodes is shown in Fig. 5. Dyes used

the present study accompanying with COOH group and thereby chelating of dyes with metal oxides was observed. It has been reported that eosin Y and mercurochrome chelate via dimmers with metal oxides[8,9].

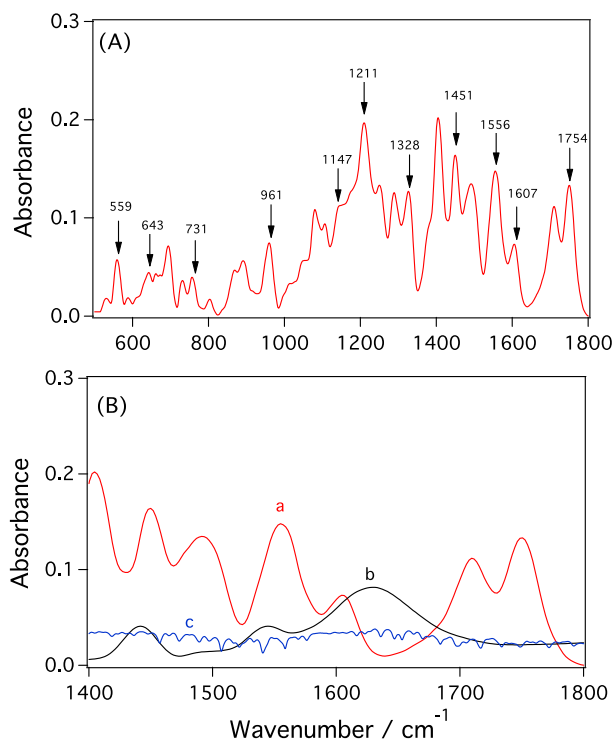
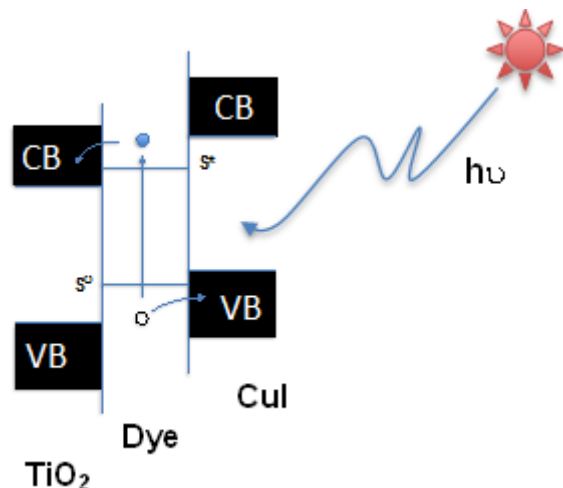


Figure 6 (A): FTIR spectrum of erythrosin B, and Fig. 6(B). FTIR spectra of (a) erythrosin B, (b) erythrosin B coated TiO_2 and (c) TiO_2

An attempt was made to measure FTIR spectra for dyes and dye coated TiO_2 samples. We were able to successfully measure FTIR of each dye. As an example, FTIR spectrum of erythrosin B powder is shown in Fig. 6(A). However, a limited number of peaks were observed for dye coated TiO_2 samples, due to trace amount of dye on TiO_2 . FTIR spectrum of erythrosin B coated TiO_2 powder is shown in Fig. 6(B). FTIR spectrum of (a) erythrosin B, (b) erythrosin B coated TiO_2 and (c) TiO_2 in the range of 1400-1800 cm^{-1} is shown in Fig. 6(B). Disappearance of peak related to C=O bond at 1754 cm^{-1} observed in the FTIR (curve b) and is confirmed of bonding of erythrosin B and TiO_2 .

Delocalized electrons in the dye molecules absorb visible light, get excited and reach their ground state via radioactive or non-radioactive processes unless they are separated efficiently. An efficient charge injection would be expected when dye molecules are in contact with semiconductors. The mechanism of charge generation in dye sensitized systems was explained by Gerischer and Tributsch, as originating from transfer of an electron by the excited dye molecule to the conduction band of the semiconductor which lies energetically lower than the excited energy level of the dye molecules [10]. Regeneration of dyes takes place by accepting an electron from the electrolyte presence in photo-electrochemical cells. Similar type of charge generation was observed for solid-state $\text{TiO}_2/\text{dye}/\text{p-semiconductor}$ (NDP) solar cells [11]. The mechanism of charge generation of NDP type solar cells is shown in scheme 2.



Scheme 2: Charge generation of TiO₂|dye|CuI type solar cell under visible light, where, CB, VB, S₀ and S* denote the conduction band, the valence band, ground state dye and excited dye, respectively.

Table 1: Parameters of the cell, open circuit voltage (V_{oc}), short circuit photocurrent (i_{sc}), fill factor (ff), efficiency (η), incident photon-to-current conversion efficiency (IPCE) for TiO₂|dye|CuI(CuSCN) cells

Cell configuration	V _{oc} / mV	i _{sc} / mAcm ⁻²	ff	η (%)
TiO ₂ MC p-CuI	523	4.15	48.8	1.06
TiO ₂ 5(6)CF p-CuI	328	3.3	36.8	0.4
TiO ₂ 4,5,6,7-TFF p-CuI	337	3	36.1	0.37
TiO ₂ RB p-CuI	295	2.8	30.6	0.25
TiO ₂ FL p-CuI	361	2.52	45.7	0.42
TiO ₂ EY p-CuI	331	1.98	25.8	0.17
TiO ₂ EB p-CuI	343	1.19	42	0.17
TiO ₂ mixture p-CuI	350	9	56.3	1.8
TiO ₂ MC p-CuCNS	534	0.75	52.3	0.21
TiO ₂ 5(6)CF p-CuCNS	406	1.25	39.7	0.2
TiO ₂ 4,5,6,7-TFF p-CuCNS	403	0.4	53.3	0.1
TiO ₂ RB p-CuCNS	400	0.44	34.6	0.1
TiO ₂ FL p-CuCNS	400	0.16	42.1	0.1
TiO ₂ EY p-CuCNS	482	0.62	45.9	0.1
TiO ₂ EB p-CuCNS	469	0.13	40.8	0.1
TiO ₂ mixture p-CuCNS	400	0.25	34.6	0.1

Photo-properties [open circuit voltage (V_{oc}), short circuit photocurrent (i_{sc}), fill factor (ff), efficiency, incident photon-to-current conversion efficiency (IPCE), power efficiency (η) for different types of TiO₂|sensitizer|p-semiconductor cells are shown in the table 1. Relatively high photocurrent observed in TiO₂|dye|CuI cells than TiO₂|dye|CuCNS cells. High photo-voltage was observed in TiO₂|dye|CuCNS cells than TiO₂|dye|CuI cells, is due to higher energy level of the conduction band of CuCNS than CuI. Among tested dyes the maximum photocurrent of 4.1 mAcm⁻² and maximum open circuit voltage of 535 mV were observed for mercurochrome sensitized solar cells. Dye coated TiO₂ electrodes absorb only a part of the solar spectrum. In this viewpoint, multi-dye systems have received much attention [12-15].

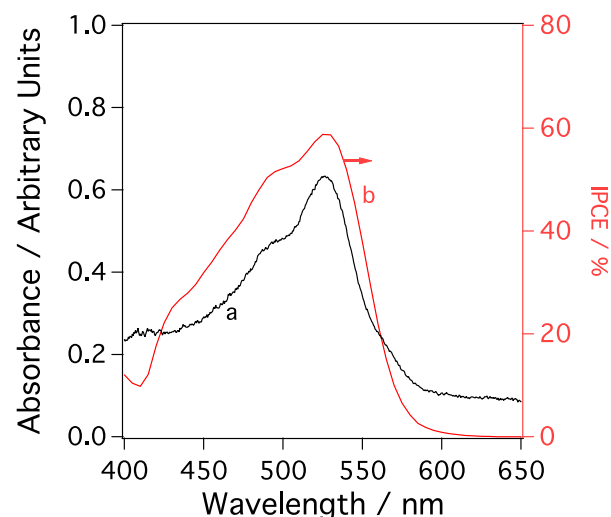


Figure 7 (a): Absorption spectrum of TiO₂|multi-dyes electrode and (b) Photocurrent action spectrum of solid-state TiO₂|dye|CuI cell with dye-multilayers.

Co-absorption of several dyes at same time makes multi-dye coated electrodes. An absorption spectrum of a solid-state TiO₂|multilayer dye electrode is shown in Fig. 7(curve a). Several peaks and humps were observed on the absorption spectrum of multi-dye coated electrode. The amount of each dye on the electrode varies with the rate constant of chelation of dyes with TiO₂ electrodes. The accurate dye amount of each dye on the TiO₂ electrode is not exactly known, at the present stage of research. Photocurrent action spectrum of a solid-state TiO₂|multilayer dye|CuI solar cell is also shown in Fig. 7(curve b). The maximum IPCE of 60% was observed for the multi-dye layer solar cell.

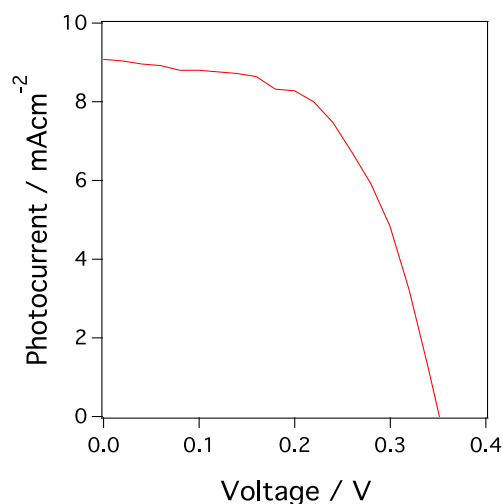


Figure 8: Current Voltage curve of solid-state TiO₂|dye|CuI cell with dye-multilayers.

Current-voltage curve of a solid-state TiO₂|multilayer dye|CuI cell is shown in Fig. 8. As is observed, dye sensitized solar cell composed with multi-dye electrode gave highest photocurrent compare to that of dyes in individually. Probably this may due to suppression of aggregation of dye molecules by others, there by suppression of dispute of energy via quenching process.

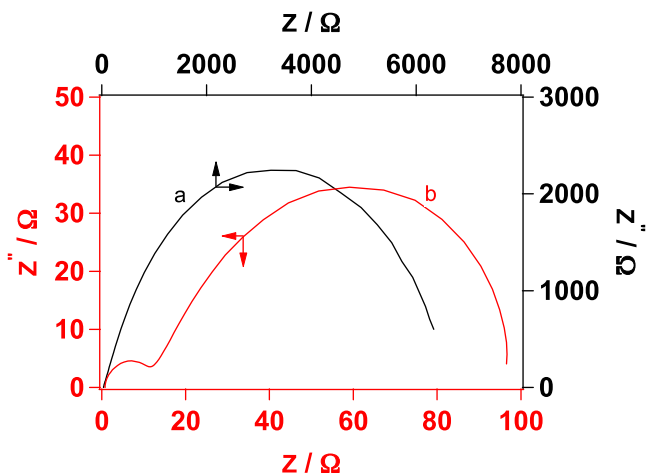


Figure 9: Impedance spectroscopy of a $\text{TiO}_2|\text{dye}|\text{CuI}$ type solar cell at natural biased (a) in dark and (b) under illumination

Figure 9 represent the impedance spectroscopy of a $\text{TiO}_2|\text{dye}|\text{CuI}$ type solar cell. A typical characteristics semi-circle was observed in Nyquist plot for the cell at natural biased in dark, which corresponds to electron transportation in the TiO_2 network and resistivity at the interfaces (curve a). Two typical characteristics semi-circles were observed in Nyquist plots for the cell at natural biased under illumination (curve b). However, the Nyquist plot significantly shifted toward the lower impedance direction and resistivity of the device become very small. This effect may be due to reduction of band bending, under illumination. The semicircle belonging to high frequencies and moderate frequencies are due to $\text{CuI}|\text{counter electrode}$ interface and to electron transportation in the TiO_2 as well as holes diffusion in the CuI network.

4. Conclusion

Dyes used in the present work have common chromophore with conjugated p-orbitals and delocalized electrons in the compounds. The chromophore is basically response to the color of dye molecules. However, our results show ligand attached chromophore also influenced the absorption properties of dyes. Van der Waals interactions make firm bonding among dye molecules and thereby exhibited interesting changes in the absorption spectra. Different types of sensitizers are used in the fabrication of a cheap solid-state cells by sandwiching sensitizer in between p-type and n-type high-band gap semiconductors.

5. Acknowledgments

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