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Comparative Study of Counter Electrodes Affecting the Conversion Efficiency in Fabrication of Dye Sensitized Solar Cell using Mixture of Dyes in Different Solvents

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In this article, dye sensitized solar cell (DSSC) was fabricated and studied using various organic dyes, which were dissolved in three solvents *viz.* ethanol, double distilled water and DMSO. Furthermore, these cells were studied using different catalytic materials as counter electrode. Differences in conversion efficiencies were observed when used with platinum, poly(3,4-ethylenedioxythiophene)-poly(styrenesulphonate)[PEDOT:PSS]-graphite and graphite as a counter electrode. Each electrons generated from dye, after its exposure to sunlight, were injected into conduction band of semiconductor (titanium dioxide). These electrons then were collected for load *via* fluorine doped tin oxide glass electrode and arrive at counter electrode, to speed up the redox reaction with tri-iodide/iodide (I_3^-/I^-) electrolyte. Firstly, high efficiencies were obtained for mixture of dyes when compared to results of their individual dyes, thus, showing the influence of mixture of dyes. Secondly varying counter electrode with photosensitizers *viz.* crystal violet B and Azur I gave conversion efficiency (η) in order platinum ≈ graphite-PEDOT: PSS > graphite.

Keywords: Counter electrode, Crystal violet B, Azur I, Poly(styrene sulphonate), Poly(3,4-ethylenedioxythiophene).

INTRODUCTION

Illuminated organic dyes can generate electricity at oxide electrodes in electrochemical cells was discovered in late 1960s [1]. The phenomenon was studied to understand and simulate the primary processes in photosynthesis at the University of California at Berkeley with chlorophyll extracted from spinach (biomimetic or bionic approach) [2]. Later, on the basis of such experiments electric power generation *via* the dye sensitized solar cell (DSSC) principle was demonstrated and discussed [3]. Grätzel & O'Regan [4] demonstrated that an appreciable quantity of electricity can be generated from sunlight by means of an inexpensive and easily produced device named DSSCs, now emerging as promising alternative to classical silicon-based solar cells; have advantages of high photocurrent conversion efficiency (IPCE), low cost and easy manufacturing technique. Highest efficiency that has been reached is 18.1% by using organic/inorganic hybrid semiconductors [5].

Metal complex sensitizers, metal-free organic sensitizers and natural sensitizers are three dye sensitizers, which are generally used in DSSCs. DSSCs that have been reported by fabricating them with many modified and synthetic organic dyes [6] are β-(ethynylbenzoic acid)-substituted push-pull porphyrins [7], organoimido-substituted hexamolybdates dyes [8], neutral, panchromatic Ru(II) terpyridine sensitizers bearing pyridine pyrazolate chelate [9], multibranched sensitizers: (a) D–(π–A)n (D = donor, π = π-spacer, A = acceptor/anchoring functionality) structures containing arylamine, carbazole, phenothiazine or phenoxazine derivatives as D groups and (b) multidonor multianchoring architectures from interconnected monobranched D–π–A arms, together with X and Y shaped dyes [10], thiophene-substituted metalloporphyrins [11], oligothiophene [12], D-π-A type indole based chromogens [13], Triphenylamine-based organic dyes [14], D–D–π-A organic dyes based on triphenylamine and indole-derivatives [15], dithienopyrrole- and fluorene-cores [16], D–π–A dyes, A1, A2-H, A2-F [17], coumarin dyes [18], porphyrin sensitizers [19], *etc*. Also, works on natural dyes as dye sensitizers [20-26] have been reported with good efficiencies.

The best photosensitization has been attained using metal transition materials [27]. Ru(II) is the most efficient dye due to its numerous advantageous features, such as good absorption, long excited-state lifetime and highly efficient metal-to-ligand charge transfer. Ru bipyridyl complexes are excellent photosensitizers due to the stability of the complexes' excited states and the long-term chemical stability of oxidized Ru(III) [28]. The standard dye used in traditional DSSCs is *tris*(2,2′-bipyridyl-4,4′-carboxylate)ruthenium(II) (N3 dye) [29]. N3 and N719 dyes contain four and two photons, respectively and were reported to absorb solar light and undergo charge transfer. Another promising candidate is tri(cyanato-2,2′2,2″-terpyridyl-4,4′,4″-tricarboxylate)Ru(II) (black dye), whose response extends approximately 100 nm further into the infrared region than the response of the N3 dye [30]. Therefore, many researchers have studied Ru-bipyridyl complexes as photosensitizers for the reactions of homogeneous photocatalytic and dye-sensitization systems. However, these complexes also have disadvantages, including high cost and the need for sophisticated preparation techniques.

When cell is placed in sunlight, dye absorb photons, causing optical excitation; the dye then injects electron in to conduction band of semiconductor (nanocrystalline-TiO_2), from where electron passes to the external circuit and reaches to counter electrode. This electron is regenerated back to the ground state by electron donation from I_3^-/I^- redox couple based liquid electrolyte [31]. This cycle continue to generate electricity. However, use of liquid electrolyte lacks the stability factor due to evaporation and leakage but also considered effective due to its mobility. Also corrosion of Pt-electrode by redox coupling reactions and degradation of dye molecules questions the commercialization on large scale. To overcome these, some measures have been applied in this study.

This work is devoted to photosensitizer and counter electrodes. Primarily, Azur A and crystal violet B dyes were individually tested in three solvents (double distilled water, ethanol and DMSO) using only platinum as a counter electrode along with KI and I_2 as an electrolyte [I^-/I_3^-] system in a liquid electrolyte such as polyethylene glycol]. Then, the efficiency of solar cell was calculated for mixture of these dyes (Azur A + crystal violet B) in different solvents as the fraction of incident power which is converted to electricity as [32]:

$$\eta = \frac{V_{oc} I_{sc} FF}{P_{in}}$$

where V_{oc} = open-circuit voltage; I_{sc}= short-circuit current; FF = fill factor; η = efficiency. P_{in} = power of incident light(W/m^2).

Current-voltage measurements (I-V measurements) took under solar spectrum to determine the efficiencies of solar cells. The I-V curves were obtained for dyes in different solvents. Result shows that the values of conversion efficiencies are affected by the solvent used for the dyes. Though the values of voltage observed were good, the dyes showed low conversion efficiency due to low current generation by the dyes.

To this end, identical method and materials were used. Later, effects of mixture of dyes by using different counter electrode on cell performance were studied. Dye sensitized solar cell (DSSC) here, is assembled using three different counter electrodes *viz*. graphite, graphite-poly(3,4-ethylenedioxythiophene)-poly(styrenesulphonate) (PEDOT:PSS) and platinum electrode on mixture of two organic dyes (crystal violet B and Azur I) dissolved in three different solvents. On comparing results of mixture of dyes (in different solvents with three different counter electrodes), it was observed that mixture of dyes were efficient with platinum and graphite-PEDOT:PSS electrode, when compared to individual dyes.

EXPERIMENTAL

Nanocrystalline TiO_2 (Anatase, Sigma-Aldrich), H_2PtCl_6 (Sigma-Aldrich), nitric acid (65%), ethanol (99.99%, Ases Chemicals), iodine (99.99%, LOBA Chemie), potassium iodide (99%, LOBA Chemie), glacial acetic acid, Triton X-100 (Fischer Scientifics), dimethyl sulphoxide (Fischer Scientifics), poly(3,4-ethylenedioxythiophene)-poly(styrenesulphonate)-(PEDOT:PSS, Sigma-Aldrich), F-doped tin oxide glass plates (conducting glass plates, surface resistivity ~10 Ω/sq, Sigma-Aldrich), polyethylene glycol (Fischer Scientifics), crystal violet B (hexamethylpararosaniline chloride), Azur I (also known as Azure B tetrafluoroborate), double distilled water and other chemicals were used as received.

F-doped tin oxide (FTO) surface resistivity: To check the surface resistivity R_s (also called as sheet resistance) of FTO conducting glass plates, the van der Pauw method [33] was used. The effect of cleaning and thermal treatment on FTO glasses thus evaluated as: R_s = (10.23 + 0.13) Ω/sq for as received, R_s = (11.57 + 0.03) Ω/sq for 99.99% ethanol washed FTOs and R_s = (10.43 + 0.07) Ω/sq for further thermal treatment (400 °C for 30 min). Thus, it ensures that calcination does not increases the sheet resistivity of FTO glass plates and is optimal for its use.

Preparation of mixture of dyes (crystal violet B and Azur I): Experiment was conducted for determining the effect of different catalytic materials on different solvents for mixture of two organic dyes. Crystal violet B and Azur I dyes were mixed and dissolved in ethanol, double distilled water and DMSO separately of same concentration (10^{-4} M). Azur I and crystal violet B dyes were also prepared individually in all the solvents of same concentration (10^{-4} M).

Fabrication of photoelectrode (DSSC assembly): TiO_2 nanoparticles (2 g) mixed with 2 mL of glacial acetic acid and stirred for 3 h. 2-3 drops of HCl were added to maintain pH 3-4; later 1 mL ethanol, 2-3 drops of Triton X-100 added in paste and stirred for 48 h at room temperature for attaining homogeneity. This paste was then applied on FTO glass plates {sheet resistance R_s = (10.43 + 0.07) Ω/sq} by doctor-blade technique. First the conductive surface of plate was checked by the use of digital multi-meter and then the edges of plate were covered with 50 μm thick scotch tape as a masking material, leaving 1 cm^2 free surfaces. A drop of the paste was poured onto one edge and was spread evenly with the help of glass rod [34]. These conductive plates with adsorbed TiO_2 paste dried at room temperature for 30 min, then after removing the scotch tape, these were transferred into electric furnace and calcined for 1 h at 500 ºC.

In the next step, the dye was allowed to adsorb on TiO_2 by placing TiO_2 film upward in petri dish filled with dye solution (covering the TiO_2 area). All the nine sets of crystal violet B + Azur I and six sets of individual dyes were prepared as mentioned above. These plates were kept in dark for 24 h and washed with the applicable solvent (solvent in which dye solution was prepared) after, to remove non-adsorbed dye. After washing, FTO conductive plates were dried in dark at room temperature and the edges were again covered carefully with scotch tape (leaving one side) to prevent it from any electrical fault.

Three types of counter electrode were prepared and examined by using three sets of Azur A, three sets of crystal violet B and three sets of crystal violet B + Azur I (different solvents) dyes. The first counter electrode *i.e.* Pt-counter electrode, was prepared simply by spreading one drop of H_2PtCl_6 solution (2 mM) on FTO glass plates, which was further calcined in electric furnace at 350-400 ºC for 30 min. Second type of counter electrode was prepared by rubbing graphite pencil on conductive surface of FTO glass plate; the third type consist of rubbing graphite pencil and applying one drop of PEDOT: PSS, which was spread by spin coating machine and later calcined at 350 ºC for 30 min.

The liquid electrolyte acts as charge mediator, prepared as follows: 0.05 M I_2 and 0.5 M KI were dissolved in polyethylene glycol and stirred well for 30 min and kept in dark bottle. This liquid electrolyte (triiodide/iodide redox couple) was applied on dye adsorbed TiO_2 plate *via* dropper and counter electrode was placed above it, leaving two edges free for connectivity. Two conducting plates were hold together using alligator clips wiping extra electrolyte off.

Cell performance: Cell performance of DSSC was evaluated by plotting graph between obtained short circuit current (I_{sc}) and open circuit voltage (V_{oc}) using digital multimeter in a circuit employed (Fig. 1). Photocurrent-voltage (I-V) measurements of DSSCs were carried out under direct sun radiation (summer, 42-45 ºC), working with similar light irradiation in all tests: 100 W/m^2 (± 5% tolerance).

RESULTS AND DISCUSSION

UV Spectrophotometer analysis: Absorption spectra of Azur I and crystal violet B dye dissolved in different solvents were obtained from ELICO-SL-244 UV-spectrophotometer. Spectra obtained were then combined to see the differences in optical properties due to solvent change.

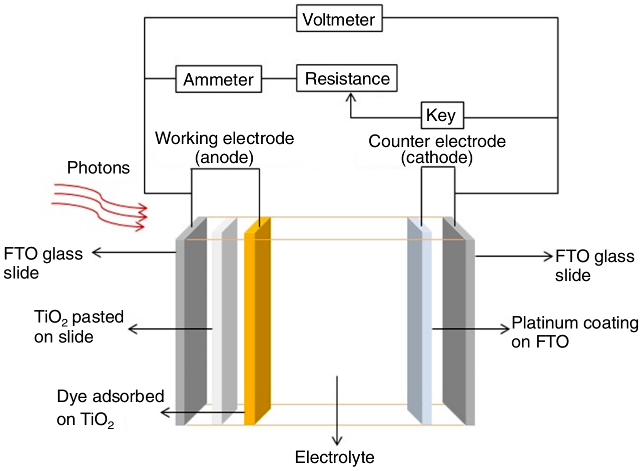

Fig. 1. Fabrication and circuit used in dye sensitized solar cell for observing open circuit voltage and short circuit current

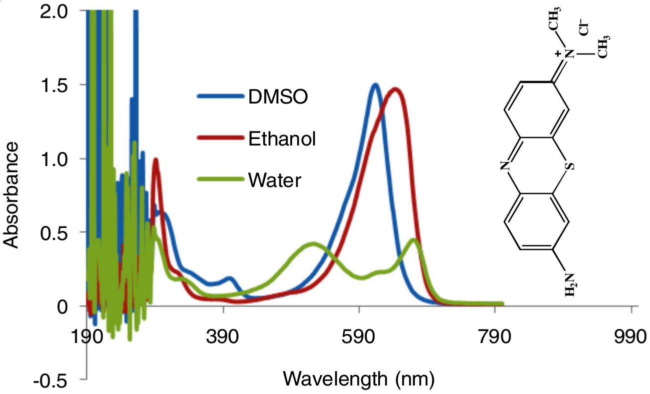

Fig. 2. UV-vis spectra of Azur I dye in different solvents

TABLE-1
OPTICAL PROPERTIES OF AZUR I IN DIFFERENT SOLVENTS

Solvent	λ_{max} (nm)	E_{max}
DMSO	614	1.4994
Ethanol	643	1.4701
Double distilled water	525 & 670	0.0326 & 0.1191

Azur I in different solvents: Azur I showed a bathochromic shift from DMSO-ethanol-water, but broad wavelength spectra was observed in double distilled water as solvent. Absorption spectrum of Azur I in three solvents explains different optical properties of photosensitizers on different solvent. As shown in Fig. 2, the spectrum range and multiple peaks are observed in double distilled water along with the highest bathochromic shift (increment in λ_{max}) *i.e.* λ_{max} of 670 nm (Table-1) yet having least absorption peak (E_{max}); and on the other hand DMSO and ethanol has only difference on their wavelength (λ_{max}) with almost equal absorption peak (Fig. 2).

Crystal violet B in different solvents: Absorption spectrum of crystal violet B shows the total opposite effect of solvent on optical properties. Unlike Azur I, crystal violet B showed maximum bathochromic shift in DMSO solvent (λ_{max} = 590 nm), though not much difference was found among all the solvents for crystal violet B dye. The spectral studies of crystal violet B showed not much difference in wavelength range for different solvents, but obtained different absorption peak; showing that it is best dissolved in ethanol and DMSO followed by water (Fig. 3). The absorption peak of crystal violet for all the three solvents *viz.* DMSO, ethanol and water were obtained at λ_{max} = 590, 583 and 579 nm, respectively (Table-2).

Crystal violet B + Azur I (mixture) in different solvents: Mixture of both dyes (crystal violet B + Azur I) in different solvents showed different results from their individual dye's result. Azur I showed highest peak in DMSO, but broad spectrum

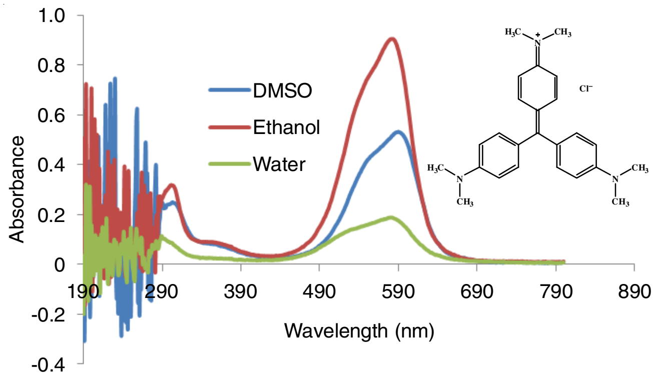

Fig. 3. UV-vis spectra of crystal violet B in different solvents

TABLE-2
OPTICAL PROPERTIES OF CRYSTAL
VIOLET B IN DIFFERENT SOLVENTS

Solvent	λ_{max} (nm)	E_{max}
DMSO	590	0.5319
Ethanol	583	0.9049
Double distilled water	579	0.1877

in double distilled water, while crystal violet B showed highest peak in ethanol with almost equal spectrum range of wavelength in all solvents. The maximum wavelength range for Azur I and crystal violet B dyes mixture found in water followed by ethanol (Fig. 4). The following graph of mixture of dyes showed maximum absorption peak at λ_{max} = 643, 640, 627 nm for water, DMSO and ethanol respectively (Table-3).

TABLE-3
OPTICAL PROPERTIES OF AZUR I AND CRYSTAL
VIOLET B DYE'S MIXTURE IN DIFFERENT SOLVENTS

Dye (photosensitizer)	Solvent	λ_{max} (nm)	E_{max}
Azur I + crystal violet B	DMSO	640	0.8216
Azur I + crystal violet B	Ethanol	627	0.3700
Azur I + crystal violet B	Double D. water	643	1.4701

Mixing dyes ensured that wavelength follows same order as Azur I dye and also good optical properties in double distilled water were obtained for mixture of dyes, fact that crystal violet B dye showed least optical properties in water.

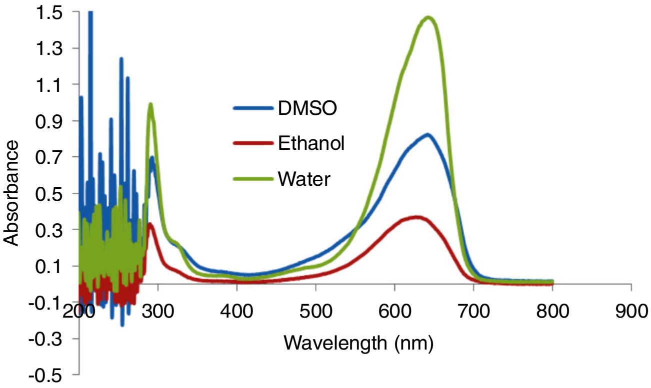

Fig. 4. UV-vis spectra of mixture of dyes (crystal violet B + Azur I) in different solvents

Scanning electron microscopy analysis: The efficiency of DSSC can also be credited to surface morphology of TiO_2 semiconducting plate. SEM helps to determine the morphology, topology and composition of material for which materials should be dry and conductive. Here TiO_2 semiconducting plate was characterized by SEM. According to principle of SEM, more the porosity, more will be the adsorption on plate, which promotes more photon adsorption *i.e.* increases the conductivity by increasing open circuit voltage (V_{oc}). Below are the images taken at magnification 7.16 KX and 412X (Fig. 5) showing the morphology of TiO_2 layer on semiconducting plate of DSSC.

DSSC characterization

Electrical parameters/I-V curves: Comparative study of individual dye (platinum as counter electrode) and mixture of dyes using different electrodes were undertaken by plotting I-V curves. The I-V curves of solar cell (Fig. 6) fabricated with mixture of dyes dissolved in double distilled water and its characteristic electrical parameters (open circuit voltage, V_{oc}; short circuit current, I_{sc}; fill factor, FF; and efficiency, η) obtained from cells with different catalytic material are shown in Table-1. Results first were obtained for individual dyes (Azur I and crystal violet B dye) and compared to mixture of dyes; low conversion efficiency was noticed for single dye system. On mixing dyes, increase in V_{oc} and I_{sc} were obtained, thus increasing

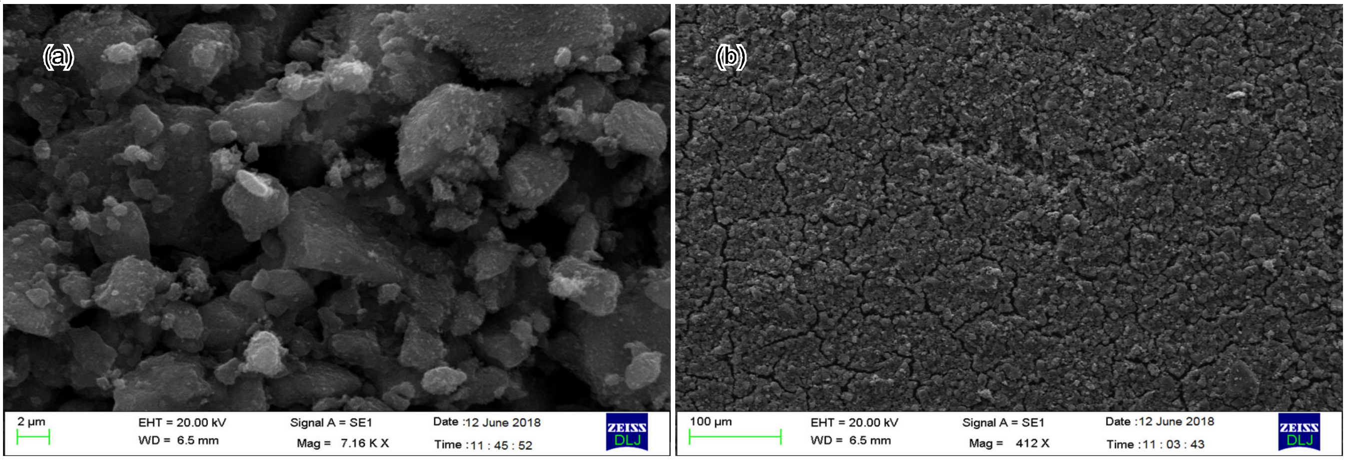

Fig. 5. SEM images of TiO_2 layer on FTO glass plates at (a) 7.16 KX and (b) 412 X

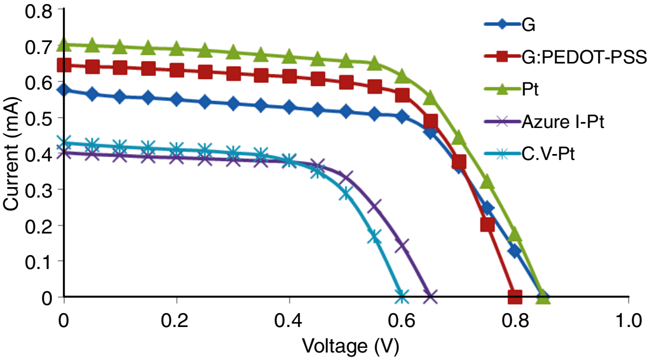

Fig. 6. I-V curves for each of the DSSC tested with different catalytic material; G-graphite; G: PEDOT-PSS-graphite + PEDOT: PSS; Pt-platinum; Azure I (in water) with platinum counter electrode; crystal violet B (in water) with platinum counter electrode

the total cell efficiency. Results of individual dyes in water as solvent for Pt-counter electrode, were labelled as reference and compared with other counter electrodes (Table-4). The highest conversion efficiency for double distilled water system was found with platinum electrode (η = 0.37%) but almost equal to this efficiency was obtained in cell with graphite-PEDOT:PSS counter electrode (η = 0.33%), which justify that instead of using single dyes (η = 0.16% and 0.15% of Azur I and crystal violet B dye, respectively) mixture of dyes can be used to increase the efficiency using low cost material such as graphite and PEDOT:PSS.

TABLE-4
ELECTRICAL PARAMETERS OF THE DSSC FABRICATED WITH DIFFERENT CATALYTIC MATERIAL (WATER SYSTEM)

CELL 1: Azur A + Crystal violet B [Solvent- double distilled water]

Type of counter electrode	I_{sc} (mA)	V_{oc} (mV)	FF	η (%)
Graphite	0.576	847	0.59	0.29
Graphite-PEDOT:PSS	0.645	807	0.63	0.33
Platinum	0.702	860	0.61	0.37
Azur I (water-Pt electrode)	0.402	625	0.66	0.16
Crystal violet B (water-Pt electrode)	0.429	573	0.63	0.15

Following same procedure as above, the experiment was conducted with two other solvents DMSO and ethanol, using three different catalytic materials, *viz.* graphite, graphite + PEDOT:PSS and platinum-H_2PtCl_6 (counter electrodes), it was observed that mixture of two dyes consistently increases the efficiency compared to Azur I and crystal violet B individually (with Pt-electrode).

In system containing ethanol as solvent, obtained I-V curve showed the lowest conversion efficiency for single dye system even with platinum as counter electrode (η = 0.06% and 0.08% of Azur I and crystal violet B respectively) (Fig. 7), justifying the effect of mixture of dyes on increasing conversion efficiency, with almost equal values obtained from platinum (η = 0.26%) and graphite-PEDOT:PSS (η = 0.23%) counter electrode (Table-5).

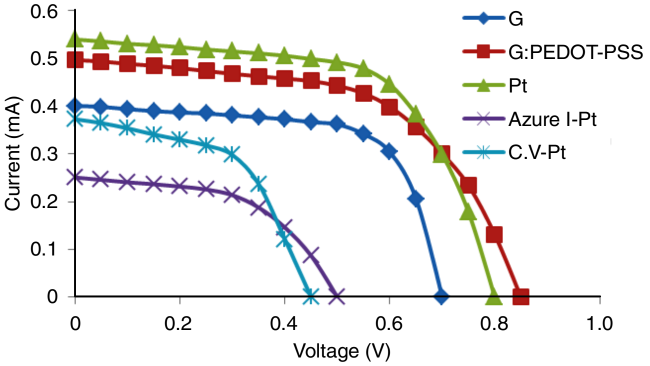

Fig. 7. I-V curves for each of the DSSC tested with different catalytic material; G-graphite; G: PEDOT-PSS- graphite+ PEDOT: PSS; Pt-platinum; Azure I (in ethanol) with platinum counter electrode; crystal violet B (in ethanol) with platinum counter electrode

TABLE-5
ELECTRICAL PARAMETERS OF THE DSSC FABRICATED WITH DIFFERENT CATALYTIC MATERIAL (ETHANOL SYSTEM)

CELL 2: Azur A + Crystal violet B [Solvent-Ethanol]

Type of counter electrode	I_{sc} (mA)	V_{oc} (mV)	FF	η (%)
Graphite	0.401	703	0.58	0.16
Graphite-PEDOT:PSS	0.497	852	0.55	0.23
Platinum	0.540	808	0.61	0.26
Azur I (ethanol-Pt electrode)	0.251	490	0.53	0.06
Crystal violet B (ethanol-Pt electrode)	0.373	449	0.53	0.08

On plotting graph between voltage and current for DMSO system, mixture of dyes showed increased electrical parameters *i.e.* I_{sc} and V_{oc} (Fig. 8) and better efficiencies than ethanol system (Table-6). Highest efficiency was obtained in cell with platinum counter electrode (η = 0.31%) followed by graphite-PEDOT:PSS (η = 0.29%) and graphite (η = 0.21%) counter electrode, which are better than single dye system.

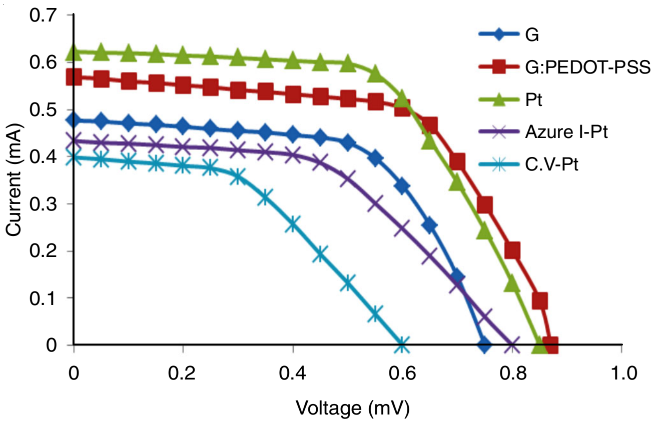

Fig. 8. I-V curves for each of the DSSC tested with different catalytic material; G-graphite; G: PEDOT-PSS- graphite + PEDOT: PSS; Pt-platinum; Azure I (in DMSO) with platinum counter electrode; crystal violet B (in DMSO) with platinum counter electrode

Azur I when tested individually with platinum electrode, conversion efficiency (η) obtained was 0.16%, 0.06% and 0.17% in double distilled water, ethanol and DMSO respectively; whereas efficiency of crystal violet B obtained as 0.15%, 0.08% and 0.10% in double distilled water, ethanol, DMSO, respec-

TABLE-6
ELECTRICAL PARAMETERS OF THE DSSC FABRICATED WITH DIFFERENT CATALYTIC MATERIAL (DMSO SYSTEM)

CELL 3: Azur A + Crystal violet B [Solvent-DMSO]

Type of counter electrode	I_{sc} (mA)	V_{oc} (mV)	FF	η (%)
Graphite	0.477	750	0.59	0.21
Graphite-PEDOT:PSS	0.569	904	0.57	0.29
Platinum	0.623	858	0.59	0.31
Azur I (DMSO-Pt electrode)	0.434	766	0.52	0.17
Crystal violet B (DMSO-Pt electrode)	0.399	574	0.47	0.10

tively, ensuring the dominancy of Azur I over crystal violet (optical properties) when both dyes were examined as mixture. Absorption spectra proves the above given data, since both absorption maxima and range were found maximum for Azur I in double distilled water and DMSO when compared to crystal violet B; whereas in ethanol, both dyes were found equally affected.

Platinum electrode thus gave nearly equal results as graphite-PEDOT: PSS gave in mixture of dyes. This is explained by broad absorption spectra which is responsible for good conversion efficiency.

Conclusion

Owing to the fact that DSSC work better with metallic dyes, many studies were performed on synthesis of various dyes incorporating metals. Previously in this study, works were based on solvent and then it was tried by mixing the dyes for better results to exclude the metallic dyes. Later this was done with different catalytic materials to decrease the cost of DSSC. Metallic dyes and all other synthesized dyes were found much expensive, thus low-cost organic dyes can be used for better performance by mixing them which increases the stability factor of dye by decreasing degradation of dye. On comparing the result, it was found that platinum electrode consistently increases the cell performance as compared to graphite, but showed results nearly equal to graphite + PEDOT:PSS, concluding that PEDOT: PSS can take over the costly platinum counter electrode with good efficiency. Increased efficiencies in all the solvents in mixture of dyes (η = 0.37%, 0.26%, 0.31% for double distilled water, ethanol and DMSO, respectively) compared to the single dye system; concludes that better cell performance can be obtained easily and at low-cost rate by mixing dyes and by mixing counter electrode [Graphite-PEDOT:PSS].

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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