

Synthesis of an amphiphilic ruthenium complex with swallow-tail bipyridyl ligand and its application in nc-DSC

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Dedicated to Michael Grätzel

Abstract

An amphiphilic swallow-tail bipyridyl ligand, 4,4'-bis(dihexylmethyl)-2,2'-bipyridine, and its heteroleptic ruthenium (II) complex were synthesized starting from dichloro-(*p*-cymene)ruthenium (II) dimer. The complex was characterized by UV/Vis and FTIR spectrophotometers, NMR spectroscopy and cyclic voltammetry. The performance of this complex as charge transfer photosensitizer in nc-TiO₂ based dye sensitized solar cells was studied under standard AM 1.5 sunlight and by using an electrolyte consisting of 0.6 M *N*-methyl-*N*-butyl imidazolium iodide (BMII), 0.1 M LiI, 0.05 M I₂, 0.5 M 4-*tert*-butyl pyridine (TBP) in acetonitrile. The complex, CS9 in DMF, gave a photocurrent density of 12.62 mA/cm², 630 mV open circuit potential and 0.62 fill factor yielding 5.68% efficiency.

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Keywords: Amphiphilic ruthenium (II) complex; Swallow-tail bipyridyl ligand; Dye sensitized solar cells

1. Introduction

Solar cells based on silicon technology are the most popular topic in the research and technology development of renewable energy sources. Although they are dominant in photovoltaic market, production of these cells still depends on complicated manufacturing and high cost processes [1]. The dye sensitized solar cells play a powerful role in research and technology development studies of renewable energy sources due to their low-cost materials and simple fabrication methods [2]. Grätzel et al. have reported the highest efficiency in nc-TiO₂ based dye sensitized solar cell (nc-DSC) with a bipyridyl ruthenium complex (black dye) [1,2]. There is not any other dye reported

in literature that could reach the efficiency of 11%. The nc-DSCs consist of photosensitizers (i.e. dyes) adsorbed on nanocrystalline semiconductor thin film electrode, electrolyte and counter electrode. The operation of nc-DSCs is different from p–n junction inorganic solar cells. The working principle of nc-DSC is based on photoelectrochemical process. The photosensitizers are excited by incoming photon at the TiO₂/electrolyte interface, and excited charge carriers, i.e. electrons, are injected into the TiO₂ conduction band. The injected electrons migrate to the working electrode, i.e. transparent conductive glass, and go to counter electrode, i.e. TCO transparent glass covered by Pt. The dye is reduced by electrolyte including redox couple or a solid state or a quasi-solid state (gel) material [3–7].

Ruthenium (II) polypyridyl complexes gain great interest because of their wide application areas. The application areas depend mainly on their octahedral geometry, ligand

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variety and controllable photophysical, photochemical and electrochemical properties [8]. Ruthenium (II) polypyridyl complexes have been utilized in nc-DSCs [6], molecular wires [9], biosensor [10] and therapeutic agent applications [11].

The amphiphilic heteroleptic sensitizers have several advantages in nc-DSC applications, such as: (i) increasing the dye loading [12]; (ii) increasing the stability of the cell [12]; (iii) providing an effective insulating barrier for back-electron transfer from TiO₂ conduction band to triiodide in the electrolyte [13], and it is a known fact that 4,4'-dicarboxy-2,2'-bipyridine presence, enhances the binding of the dye onto the nanocrystalline TiO₂ surface. Under the light of those reported determinations, but especially item (iii), we decided to synthesize a new amphiphilic heteroleptic ruthenium (II) complex that contains swallow-tail bipyridil ligand and compare its photovoltaic properties with **Z907** as the photosensitizer in nc-DSC.

2. Experimental

2.1. Materials

Dichloro(*p*-cymene) ruthenium (II) dimer, ammonium thiocyanate, 1-bromohexane, tetrabutyl ammonium hexa fluorophosphate and analytical grade solvents were purchased from Fluka. Diisopropylamine, nitric acid, acetic acid were obtained from Merck. *N*-Butyllithium (1.6 M in hexane), hydrogen hexachloroplatinate (IV) solution, ethyl cellulose, LH-20 Sephadex gel, 4,4'-dinonyl-2,2'-bipyridine and 4,4'-dimethyl-2,2'-bipyridine were provided from Aldrich. Titanium iso-propoxide was obtained from Acros. **Z907** was synthesized according to the literature [14] and all the other chemicals used were in analytical grade.

2.2. Physical measurements

UV–Vis spectra were recorded in a 1 cm path length quartz cell by using Analytic Jena S 600 UV spectrophotometer. Infrared spectra were obtained with a Perkin–Elmer, Spectrum BX-FTIR spectrophotometer, ¹H and ¹³C NMR spectra were measured on a Bruker 400 MHz spectrometer. The reported chemical shifts were against TMS. Electrochemical data were obtained by using a CH Instrument 660 B Model Electrochemical Workstation. Photovoltaic data were obtained by using Keithley 2400 source-meter and Labview data acquisition system.

2.3. Synthesis of 4,4'-dicarboxy-2,2'-bipyridine (**L1**)

Starting from 4,4'-methyl-2,2'-bipyridine, **L1** was synthesized according to the literature [15]. The yield was 96%. FTIR (KBr, cm⁻¹): 3449, 3114, 2442, 1719, 1366, 1290, 1268, 1244, 766, 682. ¹H NMR (D₂O + NaOH) δ ppm: 8,64 (d, 2H, NCHCH), 8,24 (s, 2H, CCHC), 7,73 (d, 2H, CCHCH).

2.4. Synthesis of 4,4'-bis(dihexylmethyl)-2,2'-bipyridine (**L2**)

4,4'-Bis(dihexylmethyl)-2,2'-bipyridine was prepared by modifying the procedure that has been applied to pyridine ring systems in literature to bipyridine ring [16]. A solution of butyllithium (1.6 M in hexane, 4 mmol) was added to a solution of diisopropylamine (0.2 M, 4 mmol) in dry ether at –15 °C. After stirring for 30 min, 4,4'-dimethyl-2,2'-bipyridine (1 mmol) was added dropwise. The resulting red solution was stirred for 15 min at –15 °C and then a solution of 1-bromohexane (1 M, 4 mmol) in dry ether was added. The mixture was stirred overnight at room temperature. The reaction mixture was washed with 1 M NH₄Cl solution, dried with Na₂SO₄ and evaporated to dryness. Hexane soluble part of the crude product was taken, evaporated and column chromatography (neural alumina; hexane/ether: 5/0.25) was applied. The yellow oily 4,4'-bis(dihexylmethyl)-2,2'-bipyridine was obtained as the first fraction collected from column. 57 mg, 11% yield. ¹H NMR (Fig. 1b) (CDCl₃) δ ppm: 8.58 (m, 2H, NCHCH), 8.26 (t, 2H, CCHC), 7.14 (dd, 2H, CCHCH), 2.71 (m, 2H, CCHCH₂), 1.73 (m, 8H, CHCH₂CH₂), 1.24 (m, 32H, CH₂CH₂CH₂), 0.87 (t, 12H, CH₃).

2.5. Synthesis of [RuL₁L₂(NCS)₂] (**CS9**)

Complex **CS9** was prepared according the literature [14,17] with 77% yield. FTIR (Fig. 1a) (KBr, cm⁻¹): 3411, 3024, 2931, 2856, 2102, 1716, 1560, 1405, 1234, 760. ¹H NMR (CD₃OD) δ ppm: 9.58 (d, 1H, NCHCH), 9.29 (d, 1H, NCHCH), 9.06 (s, 1H, CCHC), 8.91 (s, 1H, CCHC), 8.50 (s, 1H, CCHC), 8.32 (s, 1H, CCHC), 8.10 (d, 1H, CHCHC), 7.88 (d, 1H, NCHCH), 7.72 (d, 1H, CHCHC), 7.66 (d, 1H, CHCHC), 7.38 (d, 1H, NCHCH), 7.00 (d, 1H, CHCHC), 2.70 (2H, CCHCH₂), 1.84–1.33 (8H, CHCH₂CH₂), 1.28–1.00 (32H, CH₂CH₂CH₂), 0.87–0.78 (12H, CH₃).

Scheme 1 summarizes the synthetic strategy adopted for the preparation of **CS9**: (i) DMF, **L2**, 60 °C, Ar atmosphere; (ii) **L1**, 140 °C, Ar atmosphere; (iii) NH₄NCS, 140 °C, Ar atmosphere.

2.6. Preparation and characterization of nc-DSC

Electrically conductive oxide-coated glasses (SnO₂:F, Pilkington TEC-15; R_{sheet}: 15 Ω/□), were used as transparent electrodes. Dye sensitized mesoporous TiO₂ coated electrode was used as working electrode and platinum catalyst coated electrode was used as a counter electrode.

Anatase TiO₂ colloids were obtained from a sol–gel method including hydrolysis, autoclaving and condensation steps of titanium iso-propoxide in acetic acid solution as described in the literature [18,19]. A typical procedure for synthesis of TiO₂ nanoparticles might be described as follows: 61.7 ml of titanium iso-propoxide (98%) was added dropwise into 11.43 ml of acetic acid (100%) solution under stirring and at room temperature. Then the

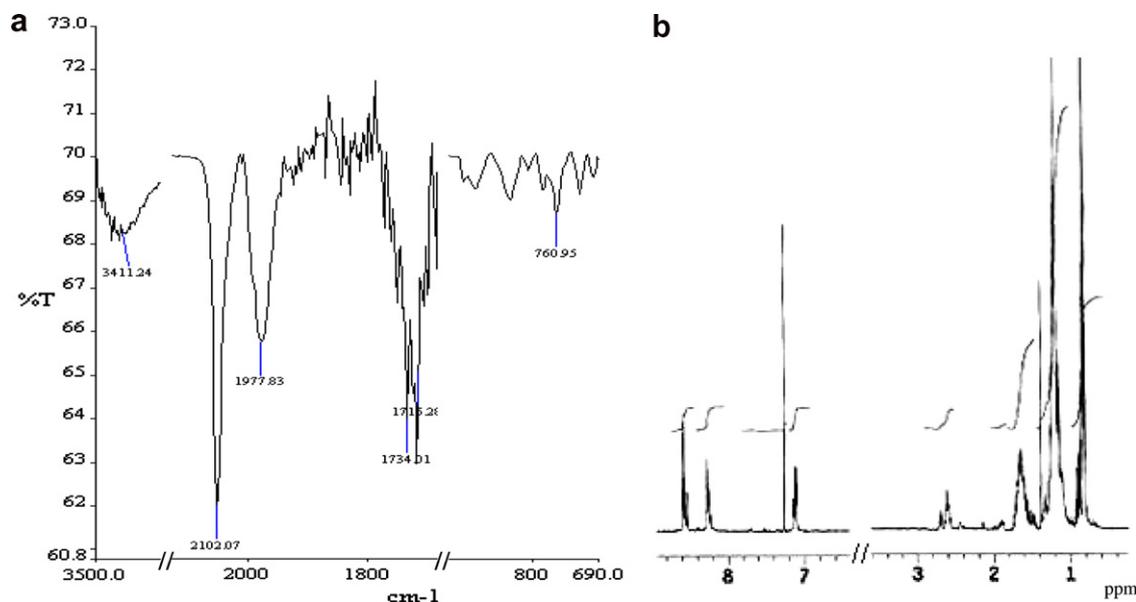
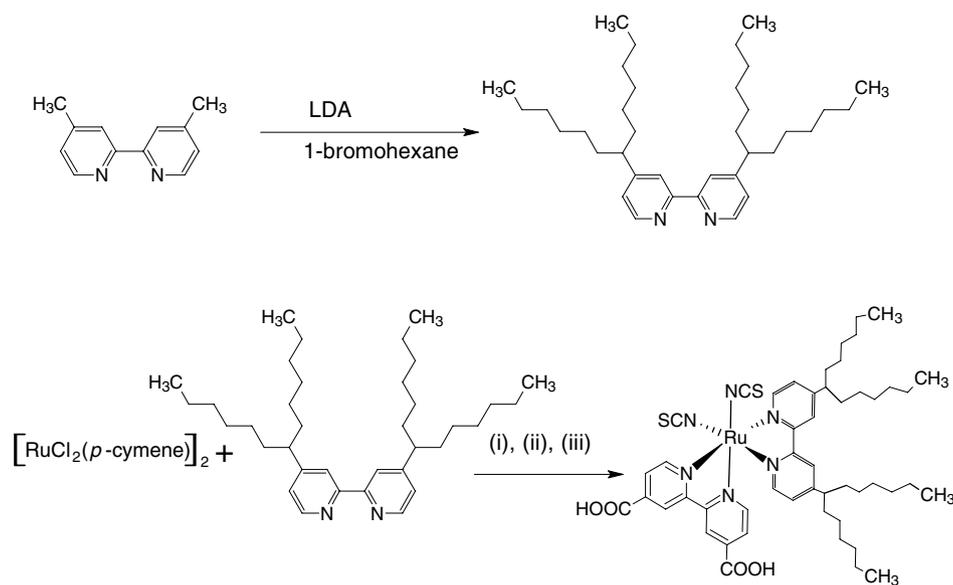


Fig. 1. (a) FTIR spectrum of CS9 complex and (b) ^1H NMR spectrum of L2 in CDCl_3 .



Scheme 1.

solution was poured into a beaker containing 290 ml of water in ice bath as fast as possible under vigorous stirring and continued stirring for an additional 30 min to complete hydrolysis. In order to achieve peptization, 5.4 ml of nitric acid (65%) was added in to the titania solution and the mixture was refluxed at 78 °C for 75 min. The volume was completed to 370 ml with water. The colloidal solution was heated for 12 h at 235 °C in a teflon baker equipped autoclave for hydrothermal growth of TiO_2 crystals. This process is called Ostwald ripening. At the end of this process TiO_2 paste was obtained with a particle size in the range of 20–30 nm. Nitric acid (2.4 ml) was added and sonicated for 20 min to disperse the agglomerates by using a titanium ultrasonic horn (100 W, 1 × 5 pulse). Then the

resultant mixture was transferred to the rotary evaporator for solvent exchange from water to ethanol and the paste was concentrated to 40% TiO_2 (w/w). 79.5 g of anhydrous terpineol and 4.5 g of ethyl cellulose were added into the TiO_2 paste for better film formation. Remaining ethanol was removed from the paste by rotary evaporator and TiO_2 colloid paste was homogenized by using a titanium ultrasonic horn.

The TiO_2 paste was coated on FTO coated glasses by screen printing technique. Coated electrodes were first dried at room temperature for 10 min and sintered at 450 °C for 30 min. Thickness of the films were measured by a Tencor Alpha Step 500 surface profiler and found as about 9.3 μm . The electrodes were dipped into dye

solutions of 5×10^{-4} M **CS9** and **Z907** in DMF and acetonitrile: *tert*-butanol (1:1) mixture for overnight. Then the electrode was kept to dry at 70 °C for 10 min.

Platinized FTO glasses were used as counter electrode. Platinization of counter electrodes were done by coating the FTO glasses with 1% solution of hydrogen hexachloroplatinate in 2-propanol and annealing at 400 °C for 30 min. *nc*-DSC was formed in sandwich geometry. A thermoplastic polymer, Surlyn-1702, which has 50 μm thickness was used as spacer and sealing material. Electrolyte was filled to the space formed between two electrodes under vacuum using a small hole pre-drilled on counter electrode. Then hole was closed by using surlyn and cover glass. The composition of electrolyte was 0.6M *N*-methyl-*N*-butyl imidazolium iodide (BMII) + 0.1 M LiI + 0.05 M I_2 + 0.5 M 4-*tert*-butyl pyridine (TBP) in acetonitrile. Active areas of the cells were defined as 0.292 cm^2 .

3. Results and discussion

3.1. Spectral and electrochemical analysis

3.1.1. Absorption and emission studies

The absorption spectra of **CS9** and **Z907** are shown in Fig. 2, and maximum absorption wavelengths and corresponding absorption coefficients are summarized in Table 1. The absorption spectrum of the fully protonated complexes were measured in DMF. The spectrum of **CS9** shows two metal-to-ligand charge transfer (MLCT) bands

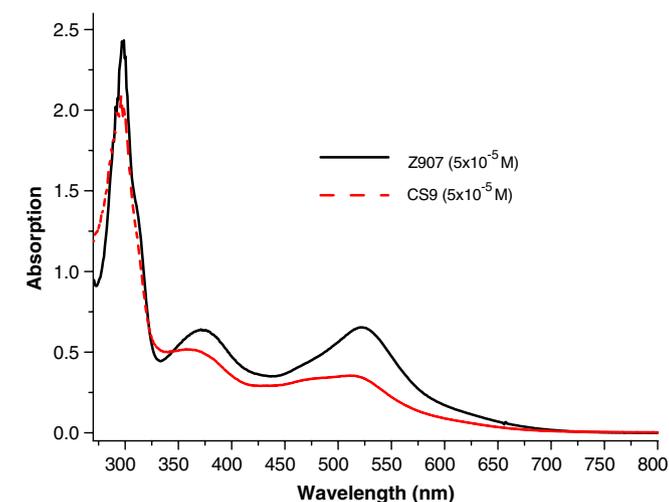


Fig. 2. UV-Vis absorption and emission spectra of 5×10^{-5} M **CS9** solution and absorption spectrum of synthesized **Z907** in DMF.

Table 1
Absorption and electrochemical data of the complexes in DMF

| Complex | $\lambda_{\text{max}}^{\text{abs}}$ (nm) ($\epsilon/10^4 \text{ M}^{-1} \text{ cm}^{-1}$) | | | E_{red1} (V) | E_{red2} (V) | E_{ox} (V) |
|-------------|---|--------------------------|------------|--------------------------|--------------------------|------------------------|
| | $\pi \rightarrow \pi^*$ | $d\pi \rightarrow \pi^*$ | | | | |
| CS9 | 296 (4.17) | 366 (1.03) | 518 (0.70) | -1.48 | -1.85 | 0.89 |
| Z907 | 297 (4.82) | 371 (1.28) | 522 (1.30) | -1.60 | -1.92 | 0.78 |

which are well-resolved at 518 and 366 nm [17,20], with a molar extinction coefficient of 7×10^3 and $1.03 \times 10^4 \text{ M}^{-1} \text{ cm}^{-1}$, respectively. The band at 295 nm is assigned to the intra ligand $\pi-\pi^*$ transition of H_2dcbpy . When compared with **Z907**, the MLCT bands of **CS9** are blue shifted approximately 4–5 nm.

3.1.2. IR spectra

The IR spectrum of **CS9** exhibits a peak at 2102 cm^{-1} which is characteristic for the *cis*-configuration of the thiocyanate group and is confirmed by the presence of vibrational band at 760 cm^{-1} (Fig. 1). The spectra shows strong band at 1716 cm^{-1} due to carboxylic acid groups. The intense peak at 1234 cm^{-1} is assigned to the $\nu(\text{C}-\text{O})$ stretch. The characteristic vibrational transitions of the alkyl chains are observed at $2856, 2931 \text{ cm}^{-1}$ [17,21].

3.1.3. Electrochemistry

The electrochemical studies were performed in a cell containing Ag/AgCl reference electrode, glassy carbon working electrode, Pt wire counter electrode and 0.1 M TBAPF6 in DMF. The redox potentials were summarized in Table 1. **CS9** shows one quasi-reversible oxidation peak and two reversible reduction peaks at +0.89, -1.48 and -1.85 V, respectively. Several consecutive voltammograms were performed and no significant change in peak currents was observed. In addition stepwise cyclic voltammograms (not shown here) were measured to check the redox process. Stepwise cyclic voltammograms shows that each redox process is separated from each other. Peak separation for oxidation of **CS9** is approximately 80 mV and for the first and second reductions of **CS9** this value is 60 mV and 70 mV, respectively. It shows that one electron process occurs for both oxidation and reduction reactions. Oxidation and reduction peaks are assigned to Ru(II) couple and 4,4'-dicarboxy-2,2'-bipyridine and 4,4'-bis(dihexylmethyl)-2,2'-bipyridine ligand, respectively [14,22]. Fig. 3 shows cyclic voltammograms of **CS9** and **Z907**, five consecutive voltammograms of **CS9** and differential of **CS9** voltammogram measured in DMF solutions.

The LUMO and HOMO energy levels are appropriate for efficient electron transfer to conduction band of anatase TiO_2 and regeneration of the dye by iodide/triiodide couple. These properties give us chance to use **CS9** as efficient sensitizer in DSCs.

3.2. *nc*-DSC performance

The efficiency of the photoelectrochemical conversion of absorbed photons was judged by measuring the IPCE% values and by the use of following equation:

$$\text{IPCE}\% = [1240 (\text{eV nm}) \times I (\text{mA}/\text{cm}^2)] \times 100 / [\lambda \times P_{\text{inc}} (\text{mW}/\text{cm}^2)]$$

where P_{inc} is the incident light power, which was measured by Nova II versatile laser power energy display at the spe-

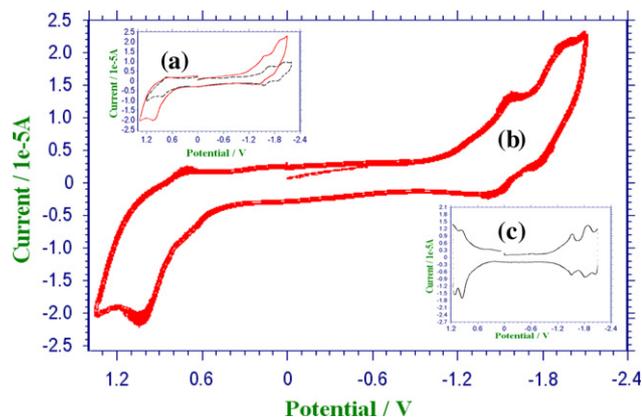


Fig. 3. (a) Cyclic voltammograms of **CS9** (solid line) and **Z907** (dashed line), (b) five consecutive voltammograms of **CS9** and (c) differential of **CS9** voltammogram measured in DMF solutions with scan speed of 250 mV s^{-1} .

cific wavelength (λ), and I is the produced photocurrent. Fig. 4 reveals that the maximum IPCE% of **CS9** is 60% where as this value is 65% for **Z907** both adsorbed on TiO_2 from DMF solutions.

Current voltage relationship of the cells were characterized by using AM 1.5 solar simulator and Keithley 2400 source-meter to obtain current–voltage (I – V) measurements. The source-meter is controlled by Labview data acquisition software. I_{sc} (mA/cm^2) is short circuit current and V_{oc} (V) is open circuit voltage. Over all efficiency defined as a ratio between output power of cell and incident light intensity;

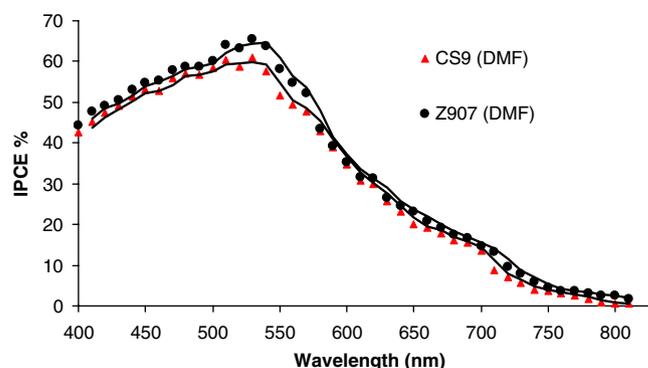


Fig. 4. Photocurrent action spectrum obtained from the cells prepared from DMF solutions of complexes, **CS9** and **Z907**.

$$\eta = P_{\max}/P_{\text{light}}$$

P_{\max} is maximum power point of cell (mW/cm^2) and P_{light} is intensity of light (mW/cm^2). Fill factor FF is formulated as

$$\text{FF} = I_m V_m / I_{sc} V_{oc}.$$

Photovoltaic performances of **Z907** and **CS9** are given in Fig. 5. The active areas of all nc-DSCs were adjusted to 0.292 cm^2 . Current density–voltage curve of dyes measured under standard test conditions (A.M 1.5, 100 mW/cm^2 light intensity at room temperature) and in dark. The photovoltaic performances of **CS9** and **Z907** are summarized in Table 2 for different solvents. The measurements are repeated several times not only by the use of the same cells but also the cells that were prepared on different dates.

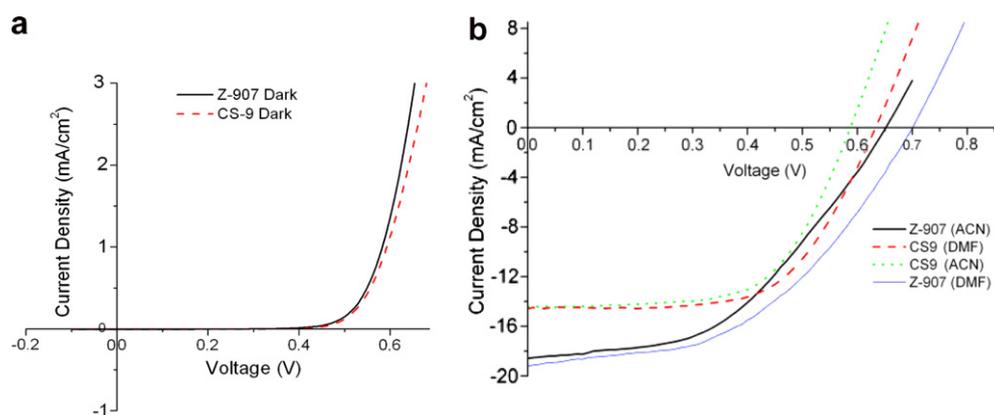


Fig. 5. I – V curve of **CS9** and **Z907** (a) in dark and (b) under 100 mW/cm^2 AM 1.5 condition at room temperature.

Table 2

I – V measurement results of nc-DSCs sensitized with **CS9** and standard dye **Z907** under illumination with 100 mW/cm^2 light intensity by AM 1.5 solar simulator

| Complex | V_{oc} (mV) | I_{sc} (mA/cm^2) | I_m (mA/cm^2) | V_m (mV) | MPP | FF | η (%) | Electrolyte | Active area (cm^2) |
|--------------------------------------|---------------|-------------------------------|----------------------------|------------|------|------|------------|-------------|-------------------------------|
| CS9 (in DMF) | 630 | 14.59 | 12.62 | 450 | 5.68 | 0.62 | 5.68 | BMII in ACN | 0.292 |
| CS9 (in ACN: <i>t</i> -BuOH) | 590 | 14.44 | 12.55 | 420 | 5.27 | 0.62 | 5.27 | | |
| Z907 (in ACN: <i>t</i> -BuOH) | 650 | 18.58 | 14.10 | 400 | 5.64 | 0.47 | 5.64 | BMII in ACN | 0.292 |
| Z907 (in DMF) | 700 | 19.18 | 14.32 | 450 | 6.44 | 0.48 | 6.44 | | |

The given values correspond to the maximum efficiencies we could obtain regularly. 14.59 mA cm⁻² short circuit current, 0.630 V open circuit voltage and 0.62 fill factor, yielding 5.68% efficiency were obtained from **CS9** cell prepared from DMF of the dye solution of **CS9**. Whereas, from acetonitrile: *tert*-butanol (1:1) solution of **CS9**, the efficiency we obtained was 5.27% (14.44 mA cm⁻², I_{sc} : 0.59 V, V_{oc} and 0.62 FF). The difference of efficiencies strongly generates from solubility difference of **CS9** in these solvents. Less solubility of the dye increases the formation of agglomerates instead of monolayer on the TiO₂ surface which creates electron traps and increases charge recombination. The reference nc-DSSC sensitized with **Z907** yields 5.64% and 6.44% conversion efficiencies from acetonitrile: *tert*-butanol (1:1) and DMF solutions, respectively. Each experimental result shows that fill factor of DSSCs sensitized with **CS9** is larger than **Z907** at the same conditions. The fill factor, essentially changes with series and shunt resistance which occurs due to internal leakages or back-electron transfer currents in nc-DSSC [23]. In our opinion, the swallow-tail alkyl chain of **CS9** exhibits better spacer properties in comparison with single chain that prevents the charge recombination of electron to electrolyte from the conduction band of TiO₂. Thus, the leakage current decreases due to the prevention the charge recombination between conduction band of TiO₂ and electrolyte. Of course to support these claim transient absorption measurements has to be performed and kinetic calculations has to be done with both of the dyes.

4. Conclusion

Here we reported the synthesis of a new amphiphilic ruthenium complex with swallow-tail bipyridyl ligand and its application in nc-DSSC. We have obtained slightly higher fill factor values with comparison to **Z907** dye under standard conditions. To claim that this new dye with a swallow-tail alkyl chain, is a good alternative for nc-DSSC application further studies have to be done. Some of these studies are in the progress and will be published soon.

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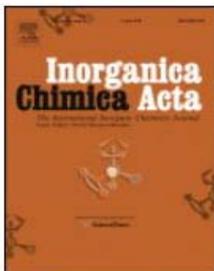
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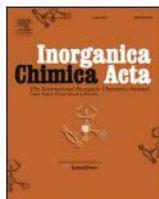
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