

Research Article

Fabrication and Characterization of Dye-Sensitized Solar Cells for Greenhouse Application

Jeum-Jong Kim,¹ Mangu Kang,¹ Ock Keum Kwak,² Yong-Jin Yoon,³
Kil Sik Min,⁴ and Moo-Jung Chu¹

¹ IT Materials and Components Laboratory, Electronics and Telecommunications Research Institute, Daejeon 305-700, Republic of Korea

² Sun Moon University, Asan-si, Chungnam 336-708, Republic of Korea

³ Department of Chemistry and Research Institute of Natural Science, Graduate School for Molecular Materials and Nanochemistry, Gyeongsang National University, Jinju 660-701, Republic of Korea

⁴ Department of Chemistry Education, Kyungpook National University, Daegu 702-701, Republic of Korea

Correspondence should be addressed to Kil Sik Min; minks@knu.ac.kr and Moo-Jung Chu; mjc@etri.re.kr

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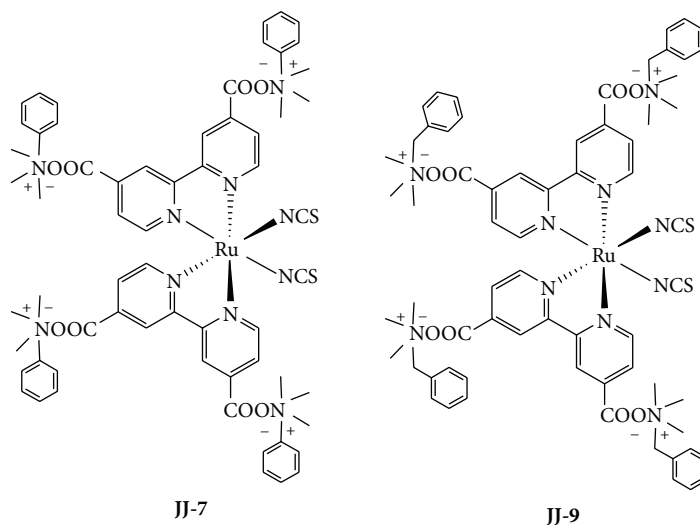
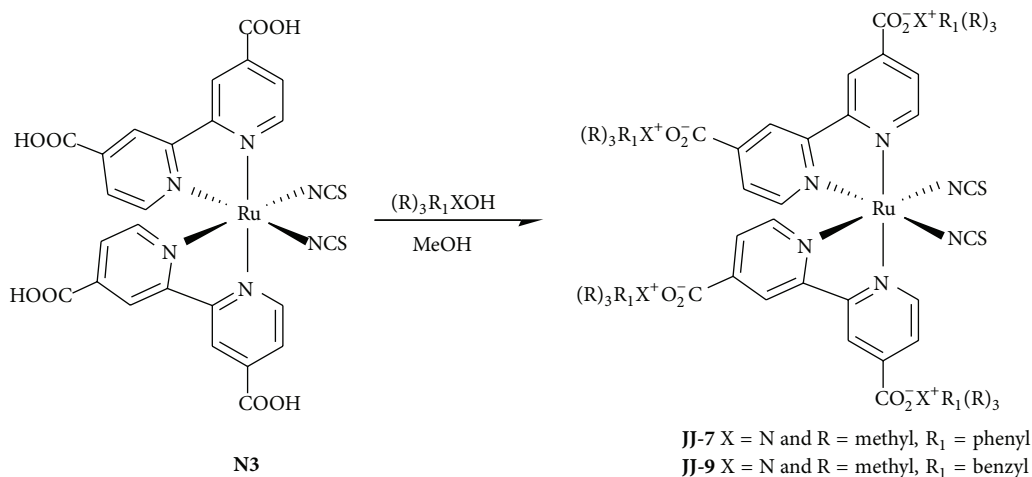
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We have developed dye-sensitized solar cells using novel sensitizers with enhanced transmittance of red (625–675 nm) and blue (425–475 nm) wavebands to control the illumination condition in the greenhouse. Novel ruthenium bipyridyl sensitizers with general formulas $(\text{Me}_3\text{PhN})_4[\text{Ru}(\text{dcbpy})_2(\text{NCS})_2]$ (JJ-7) and $(\text{Me}_3\text{BnN})_4[\text{Ru}(\text{dcbpy})_2(\text{NCS})_2]$ (JJ-9) have been synthesized and demonstrated as efficient sensitizers in dye-sensitized solar cells for greenhouse application. Under standard AM 1.5 sunlight, the solar cell of JJ-7 using a liquid-based electrolyte exhibits a short-circuit photocurrent density of 8.49 mA/cm², an open-circuit voltage of 0.83 V, and a fill factor of 0.71, corresponding to an overall conversion efficiency of 4.96% on 5 μm TiO₂ film. The transmittance of JJ-7 and JJ-9 shows 62.0% and 61.0% at 660 nm and 18.0% and 15.0% at 440 nm for cultivation on 5 μm TiO₂ film, respectively.

1. Introduction

A dye-sensitized solar cell (DSSC) is an electrochemical device that uses light-absorbing dye molecules adsorbed on semiconductor nanoparticles to generate electricity from the sunlight [1–5]. Current researches on the DSSCs are focused on the development of cell materials and manufacturing techniques that give high conversion efficiency, low cost, and stability [6–8]. The preparation of dye-sensitized solar cell for greenhouse is involved in the light manipulation for plant growth and energy-saving. The light manipulation in greenhouse is very important to improve the quantity and quality of the agricultural products. The cladding materials [9–13] and artificial lights [14–20] (LED or high-pressure sodium lamps) are used to manipulate the light in greenhouse for plant growth. Until now, no dye-sensitized solar cells (DSSCs) have been applied in greenhouse for plant growth and energy-saving. The most important wavebands for plant growth are

the absorption peaks of chlorophyll located in the red (625–675 nm) and blue (425–475 nm) regions, respectively. The red spectrum band is known to be involved in photosynthesis and the blue band is related to the photomorphogenic and the phototropic responses of plants [21]. Therefore, dye-sensitized solar cells for greenhouse can be used as technically advanced photoselective coverings that control the environmental conditions to optimize the productivity and quality of farm products and save energy. For the plant growth and energy-saving, we have focused on the development of novel sensitizers for DSSCs with enhanced transmittance of red and blue wavebands and high performance. This approach is to synthesize efficient ruthenium sensitizers through a systematic tuning of the LUMO and HOMO energy levels by introducing a ligand with a high-lying π^* molecular orbital or by stabilizing the metal t_{2g} orbital. Here, we report the synthesis of novel ruthenium(II) sensitizers (JJ-7 and JJ-9) for greenhouse DSSCs and their photovoltaic performance (Figure 1).

FIGURE 1: Molecular structures of **JJ-7** and **JJ-9**.SCHEME 1: Schematic diagram for the synthesis of sensitizers **JJ-7** and **JJ-9**.

2. Results and Discussion

The synthetic route for the preparation of **JJ-7** and **JJ-9** is depicted in Scheme 1. **JJ-7** and **JJ-9** were prepared by reaction of *cis*-dithiocyanatobis(2,2′-bipyridine-4,4′-dicarboxylate)ruthenium(II) sensitizer (**N3**) dye, trimethylphenylammonium hydroxide, and benzyltrimethylammonium hydroxide, respectively. The analytical and spectroscopic data of two sensitizers are consistent with the formulated structures.

Figures 2 and 3 show plant growth of greenhouse and their transmittance spectra using cladding materials. Plant 1 was grown under solar light and plants 2, 3, 4, and 5 were grown using IR cladding material, blue cladding material, green cladding material, and red cladding material, respectively (Figure 3). The UV-Vis spectrum of red cladding material displays transmittance band over 570 nm in visible and IR region. This band is similar to the real solar light spectrum band at red (625–675 nm) waveband. On the other hand, blue

and green cladding materials are not transmitted at red (625–675 nm) waveband (Figure 2). We have cultivated lettuce in greenhouse covered with cladding materials. The lettuce growth in red cladding material is more superior in quantity and quality to other cladding materials, but the lettuce in red cladding material does not appear red color (Figure 3). In order to obtain red color of lettuce like the real solar light, we need not only red waveband (625–675 nm) but also suitable blue (425–475 nm) waveband. Therefore, we have developed greenhouse dye-sensitized solar cells using novel sensitizers with transmittance at both red and blue wavebands.

Figure 4 shows the UV-Vis spectra of **JJ-7** and **JJ-9**, together with the **N719** absorption spectrum as a reference. The UV-vis spectrum of **JJ-7** displays two absorption bands at 380 and 514 nm, which are characteristic of the metal-to-ligand charge transfer (MLCT) bands [22, 23]. The low energy MLCT band at 514 nm of **JJ-7** is 10 nm blue-shifted relative to that of **N719** (524 nm). The band at 440 nm of **JJ-7** exhibits

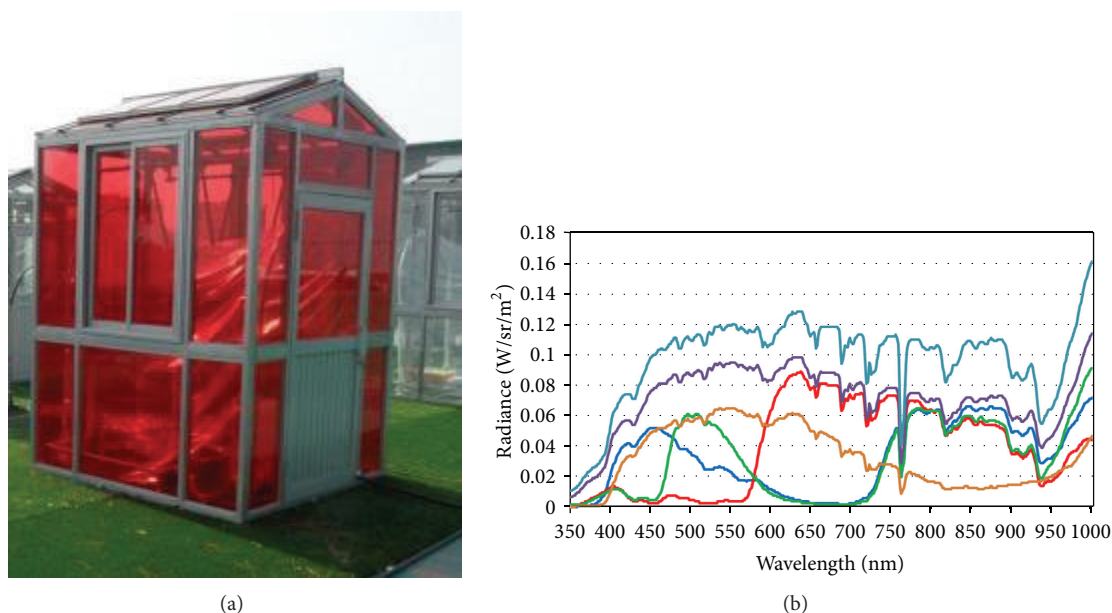


FIGURE 2: Greenhouse of plant growth and transmittance spectra under solar light (out greenhouse, dark cyan line), solar light (in greenhouse, purple line), IR cladding material (brown line), green cladding material (green line), blue cladding material (blue line), and red cladding material (red line).



FIGURE 3: Growth of plant under solar light (1), IR cladding material (2), green cladding material (3), blue cladding material (4), and red cladding material (5).

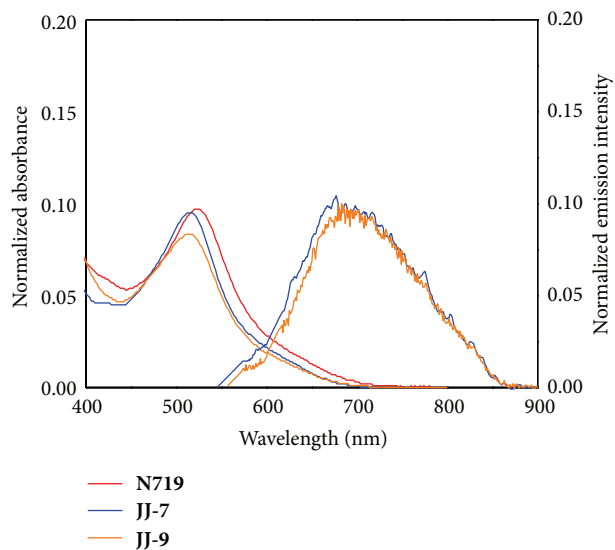


FIGURE 4: Absorption and emission spectra of JJ-7 (blue line), JJ-9 (orange line), and N719 (red line) in EtOH.

a molar extinction coefficient of $4.5 \times 10^3 \text{ M}^{-1} \text{ cm}^{-1}$, which is slightly lower than that of N719 dye ($5.4 \times 10^3 \text{ M}^{-1} \text{ cm}^{-1}$). The blue-shift and lower molar extinction coefficient are due to an increase in the energy of the LUMO of the ligand, causing the $\pi-\pi^*$ and $d\pi-\pi^*$ transitions to occur at higher energies [24]. Also, the UV-Vis spectra of JJ-9 display two absorption bands at 380 and 514 nm and a molar extinction coefficient of JJ-9 is $4.7 \times 10^3 \text{ M}^{-1} \text{ cm}^{-1}$ at 440 nm. We also observed that the sensitizers JJ-7 and JJ-9 exhibited strong luminescence maxima at 660–700 nm when they were excited with their MLCT bands in EtOH at 298 K.

The ultraviolet-visible transmittance spectra of JJ-7 and JJ-9 adsorbed on TiO_2 film are shown in Figure 5 together with the N719 transmittance spectrum as a reference. The transmittance of JJ-7 and JJ-9 on $5 \mu\text{m}$ TiO_2 film exhibits 62.0% and 61.0% at red (660 nm) and 18.0% and 15.0% at

blue (440 nm) wavelength for plant production and quality, respectively, which is higher than the corresponding value for N719 (48.0% at red (660 nm) and 7.0% at blue (440 nm) wavelength). Also, the transmittance of JJ-7 and JJ-9 on $10 \mu\text{m}$ TiO_2 film shows 62.9% and 60.5% at red (660 nm) and 7.5% and 6.3% at blue (440 nm) wavelength for plant production and quality, respectively, which is higher than the corresponding value for N719 (37.9% at red (660 nm) and 1.5% at blue (440 nm) wavelength). The higher transmittance of JJ-7 and JJ-9 compared with N719 is attributable to increased

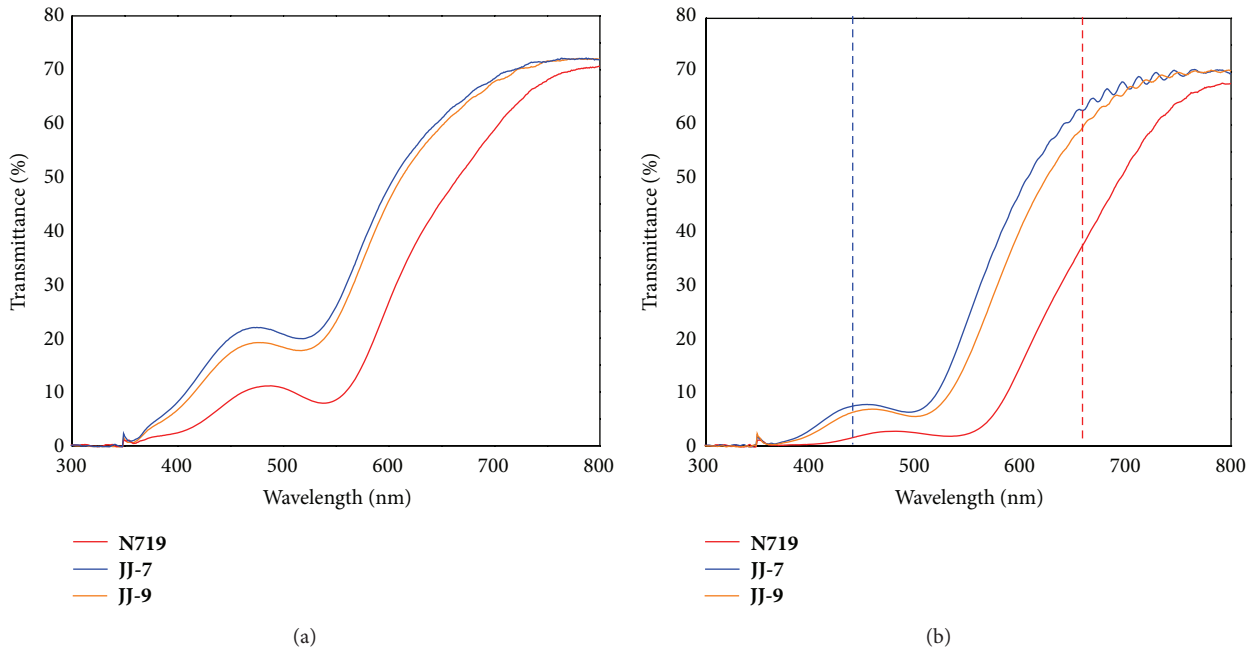


FIGURE 5: Transmittance spectra of JJ-7 (blue line), JJ-9 (orange line), and N719 (red line) (a) adsorbed on 5 μm TiO₂ film and (b) adsorbed on 10 μm TiO₂ film.

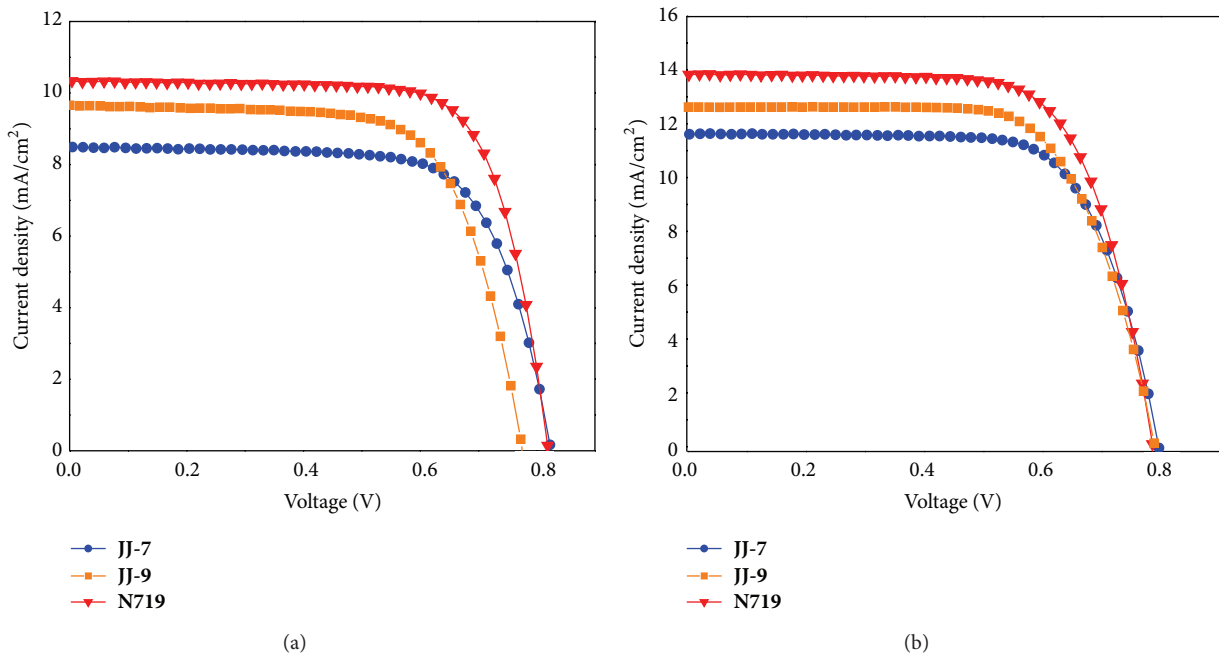


FIGURE 6: J - V curves of JJ-7 (blue line), JJ-9 (orange line), and N719 (red line) using (a) 5 μm TiO₂ film and (b) 10 μm TiO₂ film.

HOMO-LUMO energy gaps by electron-withdrawing abilities in a ligand and low molar extinction coefficient.

The J - V curves for the devices based on JJ-7 and JJ-9 are shown and compared with those of N719 in Figure 6. Under standard global AM 1.5 solar conditions, when 5 μm TiO₂ film was used, the JJ-7 and JJ-9 sensitized cell gave a short circuit photocurrent density (J_{sc}) of 8.49 and

9.40 mA cm⁻², an open circuit voltage (V_{oc}) of 0.83 and 0.78 V, and a fill factor (FF) of 0.71 and 0.69, corresponding to overall conversion efficiency (η) of 4.96% and 5.07%, respectively (Table 1). Under the same condition, the N719 sensitized cell gave a J_{sc} of 10.34 mA cm⁻², a V_{oc} of 0.82 V, and a FF of 0.74, corresponding to η of 6.25%. When 10 μm TiO₂ film was used, the JJ-7 and JJ-9 sensitized cell gave short circuit

TABLE 1: Optical, redox, and DSSC performance parameters of dyes.

Dye	$\lambda_{\text{abs}}^{\text{a}}/\text{nm}$ & $\lambda_{\text{trans}}^{\text{b}}/\text{nm}$ (%)	$E_{\text{redox}}^{\text{c}}/\text{V}$	$E_{0-0}^{\text{d}}/\text{V}$	$E_{\text{LUMO}}^{\text{e}}/\text{V}$	J_{sc} (mA cm^{-2}) ($5/10 \mu\text{m}$)	V_{oc} (V) ($5/10 \mu\text{m}$)	FF ($5/10 \mu\text{m}$)	η^{f} (%) ($5/10 \mu\text{m}$)
JJ-7	380, 514 & 440 (18), 660 (62)	1.07	2.08	-1.01	8.49/11.68	0.83/0.80	0.71/0.71	4.96/6.58
JJ-9	380, 514 & 440 (15), 660 (61)	1.03	2.06	-1.03	9.40/12.62	0.78/0.80	0.69/0.69	5.07/6.93
N719	382, 524 & 440 (7), 660 (48)		2.03 ^g (1.97 ^h)		10.34/13.87	0.82/0.79	0.74/0.70	6.25/7.67

^aAbsorption spectra were measured in EtOH solution. ^bTransmittance spectra were measured on 1-layer TiO₂ film. ^cRedox potential of dyes on TiO₂ was measured in CH₃CN with 0.1 M (*n*-C₄H₉)₄NPF₆ as a scan rate of 100 mV s⁻¹ (vs. NHE). ^d E_{0-0} values were estimated from the onset of absorption spectrum. ^e E_{LUMO} was calculated by $E_{\text{ox}} - E_{0-0}$. ^fPerformances of DSSCs were measured with 0.18 cm² working area. Electrolyte: 0.6 M DMPImI, 0.05 M I₂, 0.5 M TBP, and 0.1 M LiI in acetonitrile. ^gEnergy level calculated by experiment. ^hEnergy level calculated at the B3LYP/3-21 G.

photocurrent density (J_{sc}) of 11.68 and 12.62 mA cm⁻², an open circuit voltage (V_{oc}) of 0.80 and 0.80 V, and a fill factor (FF) of 0.71 and 0.69, corresponding to overall conversion efficiency (η) of 6.58% and 6.93%, respectively (Table 1). Under the same condition, the **N719** sensitized cell gave a J_{sc} of 13.87 mA cm⁻², a V_{oc} of 0.79 V, and a FF of 0.70, corresponding to η of 7.67%. A slightly lower J_{sc} of **JJ-7** and **JJ-9** relative to **N719** can be related to the increase of transmittance and the sparse packing of the **JJ-7** and **JJ-9** monolayers on the TiO₂ electrodes. To clarify the above explanations, we measured the amount of dyes adsorbed on TiO₂ film by desorbing the dyes from the TiO₂ surface with KOH. The amounts of three dyes adsorbed on TiO₂ film were measured to be 2.94×10^{-7} , 3.02×10^{-7} , and 3.76×10^{-7} mmol cm⁻² for **JJ-7**, **JJ-9**, and **N719**, respectively. The low adsorption of **JJ-7** and **JJ-9** can be due to the presence of bulky protecting groups with electron withdrawing abilities of tetra-substituted ammonium groups and the electrostatic repulsion of negatively charged carboxylic groups.

The electrochemical properties of the two sensitizers **JJ-7** and **JJ-9** were studied by cyclic voltammetry in CH₃CN with 0.1 M tetrabutylammonium hexafluorophosphate using TiO₂ film with adsorbed dyes as working electrode. The oxidation potentials of **JJ-7** and **JJ-9** adsorbed on TiO₂ film show quasi-reversible couples at 1.07 V and 1.03 V versus NHE, respectively (Table 1). The Ru^{III/II} oxidation potentials of **JJ-7** and **JJ-9** are more positive than that of **N719**. The HOMO-LUMO energy band gaps (E_{0-0}) of **JJ-7** and **JJ-9** determined from the intersection of absorption and emission spectra are 2.08 and 2.06 eV, respectively, more increased than that of **N719** (1.85 eV for experimental calculation [25] and 1.97 eV for theoretical calculation by B3LYP/3-21 G [26, 27]). This reflects the increased electron withdrawing properties of ligand with tetra-substituted ammonium groups. The reduction potentials of two dyes calculated from the oxidation potentials and the E_{0-0} determined from the intersection of absorption and emission spectra are -1.01 V for **JJ-7** and -1.03 V for **JJ-9** versus NHE. A negative shift in the reduction potential of **JJ-7** and **JJ-9** compared to **N719** is attributable to electron-withdrawing abilities in ligands and increased HOMO-LUMO energy band gaps.

Figure 7 shows intensity of radiation in dye-sensitized solar cell using **JJ-7** and **N719** sensitizers. After making box,

we have measured the intensity of radiation as in Figure 7. The radiation intensities for **JJ-7** sensitizers with both 5 and 10 μm TiO₂ thickness films are much higher than that of **N719** sensitizer with 5 μm TiO₂ thickness film. This result demonstrates that the novel **JJ-7** sensitizer is very effective in greenhouse dye-sensitized solar cell to grow plant. On the other hand, the existing **N719** sensitizer is not useful because of the low quantity of light. The different light quantity might be caused by the different molecular structure of the dyes. The high quantity of light in **JJ-7** compared to that of **N719** may be due to the defects of adsorption of **JJ-7** sensitizer on the TiO₂ electrodes.

In conclusion, two novel ruthenium bipyridyl sensitizers have been synthesized and characterized. A solar-to-electricity conversion efficiency of 4.96% (for 5 μm TiO₂ film) and 6.58% (for 10 μm TiO₂ film) for **JJ-7** is comparable to 6.25% (for 5 μm TiO₂ film) and 7.67% (for 10 μm TiO₂ film) for the **N719**-sensitized solar cell. The high transmittance (62% at 660 nm and 18% at 440 nm) of **JJ-7** is attributed to its low absorption extinction coefficient of MLCT band in the visible region and blue-shift by an increased HOMO-LUMO energy band gap. We believe that the development of highly efficient sensitizers for greenhouse dye-sensitized solar cell is possible through meticulously molecular engineering, and work on these is now in progress.

3. Experimental Section

3.1. Fabrication of Dye-Sensitized Solar Cells. Fluorine-doped tin oxide (FTO) glass plates (Pilkington TEC Glass-TEC 8, solar 2.3 mm thickness) were cleaned in a detergent solution using an ultrasonic bath for 30 min and then rinsed with water and ethanol. Then, the plates were immersed in 40 mM TiCl₄ (aqueous) at 70°C for 30 min and washed with water and ethanol. A transparent nanocrystalline layer was prepared on the FTO glass plates by using a doctor blade printing TiO₂ paste (Solaronix, Ti-Nanoxide T/SP), which was then dried for 2 h at 25°C. The TiO₂ electrodes were gradually heated under an air flow at 325°C for 5 min, at 375°C for 5 min, at 450°C for 15 min, and at 500°C for 15 min. The thickness of the transparent layer was measured by using an Alpha-step 250 surface profilometer (Tencor Instruments, San Jose, CA). The resulting film was composed of a 5

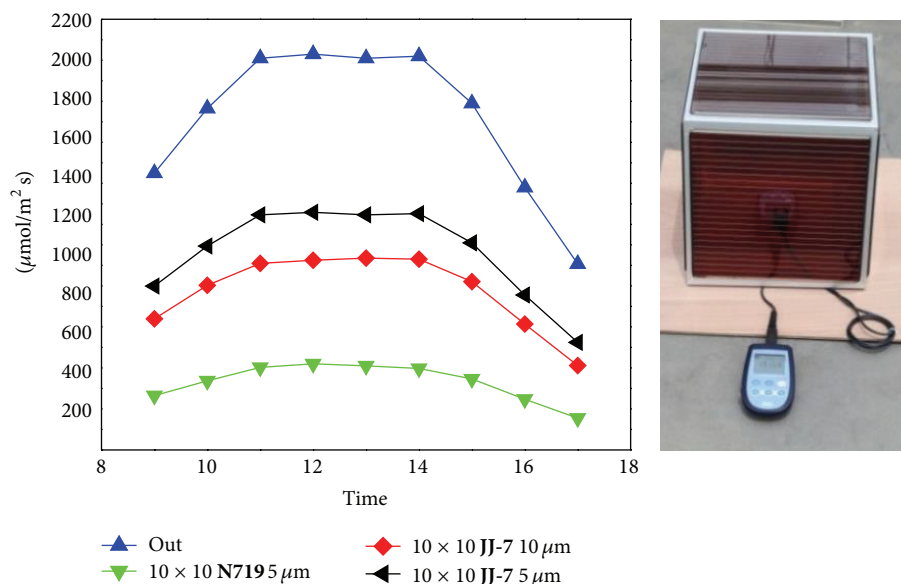


FIGURE 7: The intensity of radiation for JJ-7 (5 μm , black line), JJ-7 (10 μm , red line), N719 (5 μm , green line), and out (blue line) and greenhouse test-bed of dye-sensitized solar cell using new sensitizer (JJ-7).

and 10 μm thick transparent layer. The TiO_2 electrodes were treated again with TiCl_4 at 70°C for 30 min and sintered at 500°C for 30 min. Then, they were immersed in JJ-7 and JJ-9 (0.3 mM in ethanol) solutions and kept at room temperature for 24 h. FTO plates for the counter electrodes were cleaned in an ultrasonic bath in H_2O , acetone, and 0.1 M aqueous HCl, subsequently. The counter electrodes were prepared by placing a drop of an H_2PtCl_6 solution (2 mg Pt in 1 mL ethanol) on an FTO plate and heating it (at 400°C) for 15 min. The dye adsorbed TiO_2 electrodes and the Pt counter electrodes were assembled into a sealed sandwich-type cell by heating at 80°C using a hot-melt ionomer film (Surlyn) as a spacer between the electrodes. A drop of the electrolyte consisting of 0.6 M 1,2-dimethyl-3-propylimidazolium iodide (DMPII), 0.05 M I_2 , 0.1 M LiI, and 0.5 M *tert*-butylpyridine in acetonitrile was placed in the drilled hole of the counter electrode and was driven into the cell via vacuum backfilling. Finally, the hole was sealed using additional Surlyn and a cover glass (0.1 mm thickness).

3.2. Typical Procedures and Analytical Data

JJ-7 Complex. A mixture of N3 (100 mg, 0.141 mmol) and trimethylphenylammonium hydroxide (434 mg, 0.708 mmol) in MeOH (2 mL) was stirred at room temperature for 2 h. The pure product JJ-7 was obtained by Sephadex LH-20 column with methanol as eluent. Yield: 97%. ^1H NMR (CD_3OD): 9.43 (d, 2H, $J = 5.7$ Hz), 8.90 (s, 2H), 8.74 (s, 2H), 8.13 (dd, 2H, $J = 5.7$ Hz), 7.93 (m, 6H), 7.63–7.50 (m, 18H), 3.70 (s, 36H). Anal. calcd for $\text{C}_{62}\text{H}_{68}\text{N}_{10}\text{O}_8\text{RuS}_2$: C, 59.74; H, 5.50. Found: C, 59.77; H, 5.48.

JJ-9 Complex. A mixture of N3 (100 mg, 0.141 mmol) and benzyltrimethylammonium hydroxide (296 mg,

0.708 mmol) in MeOH (2 mL) was stirred at room temperature for 2 h. The pure product JJ-9 was obtained by Sephadex LH-20 column with methanol as eluent. Yield: 94%. ^1H NMR (CD_3OD): 9.41 (d, 2H, $J = 5.8$ Hz), 8.91 (s, 2H), 8.75 (s, 2H), 8.10 (dd, 2H, $J = 5.8$ Hz), 7.54 (m, 24H), 4.55 (s, 8H), 3.12 (s, 36H). Anal. calcd for $\text{C}_{66}\text{H}_{76}\text{N}_{10}\text{O}_8\text{RuS}_2$: C, 60.86; H, 5.88. Found: C, 60.78; H, 5.94.

Conflict of Interests

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

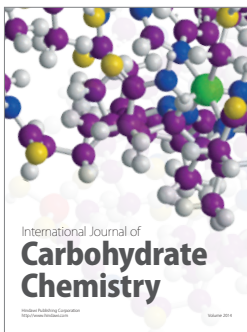
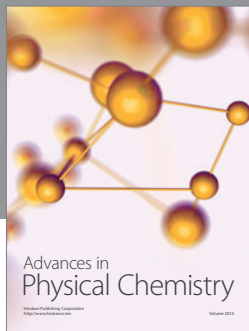
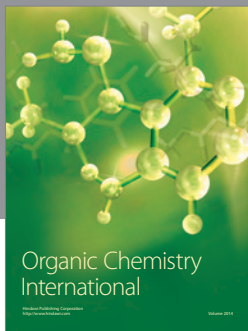
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