Special Feature: Organic Materials

Chemical Stability of Sensitizer Molecules for Dye Sensitized Solar Cells

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■**ABSTRACT**‖   The chemical stability of sensitizer molecules for dye sensitized solar cells (DSCs) was reviewed. Widely used Ru complex dyes have extremely high turnover (in case of N3 dye, 10^7). For further improvement of the long-term durability of Ru dyes, the decomposition pathway, i.e., the photochemical or chemical decomposition of the NCS group, must be avoided. The interaction between a dye and its surrounding environment such as TiO_2 (hydrophobicity at the surface) and electrolyte (iodide) is a factor determining the durability of the DSC cell. For DSCs sensitized with organic dyes, knowledge of the degradation mechanism, stability and long-term durability is limited. Our study on the stability of a DSC sensitized with a typical donor-acceptor type organic dye (D131) revealed the degradation mechanism of the dye in the DSC cell. The result suggests that in the 85°C-dark condition, degradation of the dye is promoted by iodine and amine in the electrolyte. Under the light irradiation condition, dye decomposition was suggested to start from an unstable oxidized state formed after the electron injection. By model reaction analysis and analysis of decomposition in the cell, the decomposition product of D131 was found to be decarboxylated D131, which has lost the carboxylic acid group that is indispensable for absorption on TiO_2. The liberation of the D131 moiety from the electrode by the decomposition is found to be a major degradation mechanism.

■**KEYWORDS**‖   Dye Sensitized Solar Cells, Dye, Sensitizer, Degradation Mechanism, Durability, Ruthenium, Red Dye, Organic Dye, Decarboxylation, Electrolyte

1. Introduction

It is predicted that world energy consumption will grow by 53% from 2008 to 2035 due to global economic growth.[1] Massive energy consumption will cause problems such as the depletion of fossil resources, global warming and its associated climate change, and strongly urges us to develop environmentally sustainable energy technologies. Among these renewable energy technologies, photovoltaic conversion of solar energy is considered as a promising candidate because of its clean, renewable (unlike gas, oil and coal) and sustainable nature. In 1991, dye-sensitized nanocrystalline TiO_2 solar cells (DSCs) based on the mechanism of a fast regenerative photoelectrochemical charge separation process between dye sensitizer and TiO_2 were first reported by Grätzel et al.[2] The overall efficiency of this new type of solar cell was 7.1–7.9% (under simulated solar light), which is comparable to that of amorphous silicon solar cells,[3] and in recent years it has reached more than 10% under AM 1.5 (1000 W/m²).[4-6] DSCs have been extensively studied for their attractive advantages over silicon based solar cells, i.e., their inherent low module cost, easy fabrication process, light weight, and flexibility.[7-10]

In DSCs, a dye molecule is an indispensable component to generate electricity by absorbing sunlight then exiting and ejecting the HOMO (highest occupied molecular orbital) electron into TiO_2. To attain the high photovoltaic efficiency of DSCs, the dye molecule should have a high absorption coefficient over a wide range of wavelengths, and transfer electrons fast and highly efficiently. Many dyes have been reported to improve the performance of DSCs. For example, Ru complex dyes originating from "red dye" [*cis*-(2,2'-bipyridyl-4,4'-dicarboxylate)$_2$Ru(NCS)$_2$, (N3), **Fig. 1**] by Grätzel[2] have been studied in detail and "non-metallic" organic dyes such as indoline dye (D149)[11] are known as highly efficient dyes and their structures have been intensively modified because their colors are tunable by changing the structures.

Furthermore, to make DSCs with long-term durability, the dye sensitizer should be stable not only to the redox cycle through electron exchange among

TiO$_2$ and electrolyte, but also to thermal and photochemical stress over many years. Although organic materials that form elaborate dye sensitizers are recognized generally to be not very stable to high temperature, highly oxidative, reductive or light irradiation conditions, all are inevitable conditions under which DSCs operate. So, in spite of its importance, it is still a tough challenge to make robust dye sensitizers.

In this paper, we focus our attention on the chemical stability of sensitizer molecules for dye sensitized solar cells. First, stability research on the most widely used Ru complex dyes and their DSCs is reviewed, and then our research on the degradation mechanism of an organic dye sensitizer is reported as an example of a chemically stable organic dye.

2. Stability of Ru Complex Dye

The first reported dye molecule for DSCs was the trinuclear ruthenium complex by Grätzel[2] (Fig. 1). Even in the earliest research, cell durability was tested and over 90% cell performance was maintained after 2 months of light soaking, which indicated potential high durability of Ru dyes. Since then, Grätzel's group has presented results of high durability every time they report new dyes.[12] In fact they report that turnover of the N3 dye[13] in the cell is extremely high, 10^7, which corresponds to a few years of cell lifetime.

For further improvement of the durability of dyes, it is important to understand the degradation process of the dye. However, methods to analyze the structural change of dye molecules during the degradation process in a cell are limited to a few techniques: laser Raman spectroscopy or IR spectroscopy with special infrared transmitting cells. For this reason, degradation reactions have been investigated in combination with model reactions.
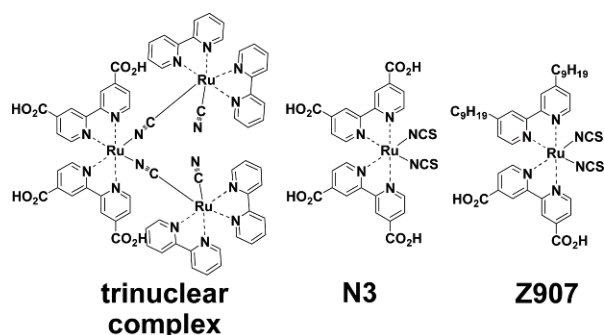
Grätzel et al. found the transformation of the NCS ligand in the N3 dye into a CN group by irradiation in methanol or chemical oxidation in water[14] (**Fig. 2**). Although it is not clear where the sulfur has gone, they suggest that the degradation mechanism is oxidation of Ru(II) in N3 to Ru(III) and electron transfer between Ru(III) and NCS promoted by photoirradiation or chemical oxidation. This structural change causes a blue shift of the spectrum, which would affect the efficiency. Additionally a ruthenium NCS complex [Ru(bpy)$_2$(NCS)$_2$, (bpy: 2,2'-bipyridine)] loses NCS by photochemical dissociation in DMF (*N,N*-dimethylformamide). The quantum yield of this elimination reaction is 7×10^{-4}, which is not negligible considering chemical stability of dyes for long-term durability tests.

Since Ru dye on TiO$_2$ generates electric power in electrolytes, in which a large molar excess to Ru dye exists, the possibility of a reaction between Ru dye and constituents of the electrolyte (iodine, iodide, amine, solvent, etc.) must be considered. In fact, N3 dye displaces the NCS ligand with the iodide in alcohol.[14] These results from model reactions show that the NCS group in N3 dye has a tendency to decompose or be displaced.

The chemical stability of dye molecules in photovoltaic cells was also studied. Grätzel mentioned that the NCS group of a Ru complex increases stability against decomposition to CN and displacement with iodide when the Ru complex is on the TiO$_2$[14]. Rapid reduction of the unstable Ru(III) state by the redox system in the electrolyte inhibits the decomposition

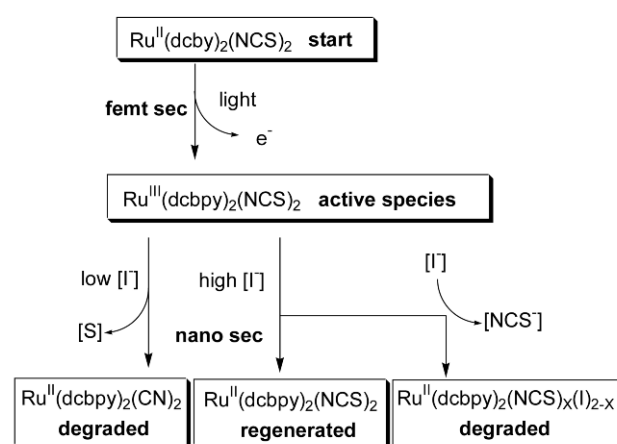**Fig. 1**   Structure of Ru complex dyes.

Fig. 2   Degradation mechanisms of N3 dye (dcbpy: 2,2'-bipyridyl-4,4'-dicarboxylate).

pathway from the unstable Ru(III) state. The reduction speed is estimated to be 10^8-10^9 faster than the decomposition reactions.

Hagfeldt et al.[15] studied in situ Raman spectroscopy of the NCS group of red dye in the DSC cell. They found that the intensity of NCS scattering decreased as the iodide concentration in the electrolyte increased, and at the typical iodide concentration for DSC cells, no NCS scattering was detected. Displacement of NCS by iodide in the complex was inferred by the wavelength shift of the absorption spectrum. By their experiment, t-butylpyridine (TBP) in the electrolyte was found to slow the NCS substitution reaction presumably by depression of the reactivity of iodide with NCS by complex formation of iodine with TBP.

On the other hand, Kato et al. performed long-term outdoor durability tests of DSCs with N719 (tetrabutylammonium salt of N3) as a sensitizer over two and half years.[16] They compared the Raman scattering intensities of the NCS group and the carboxybipyridine ligand in the dye complex before and after the test and found no significant intensity change during the test.

As mentioned above, the difficulty in investigating the internal state of a dye in the cell and the difference in the contents of the cells among researchers might make some difference in the results. In any case, these results suggest that we must consider not only the chemical stability of the dye itself, but also the interaction between the dye and the environment surrounding the dye such as TiO_2 and electrolyte.

The dye structure also affects the durability. Z907, which is an alkyl chain substituted derivative of N719 (Fig. 1), was reported to improve the durability and resistance to water of the cell due to the suppression of dye elimination from the TiO_2 surface by the high hydrophobicity of the electrode modified with Z907.

3. Stability of Organic Dye

In contrast to Ru complex dyes, "non-metallic" organic dyes do not have ligands that could potentially cause elimination. However, organic dyes might change their light absorption, energy level or TiO_2 adsorption characteristics by organic reactions. Especially, it should be noted that TiO_2 (anatase) is known as a photocatalyst to decompose organic materials.[17] Thus it is not hard to anticipate that the cell environment would be tough for an organic dye. However, little is known about the degradation

mechanism, stability and long-term durability of DSCs sensitized with organic dyes. Since a large variety of organic dye structures have been reported and there are a variety of decomposition modes, it will be difficult to present a generalized decomposition scheme for an individual structure at this time. Therefore, we picked one typical DA type organic dye, D131, and studied its detailed decomposition behavior to understand the decomposition mechanism of organic dyes.[18,19] D131 is a yellow indoline dye which absorbs light at a shorter wavelength than the red dye (N719) (**Fig. 3**). D131 has an aromatic amine (which is a part of the indoline ring) as a donor and a cyanoacrylate as an acceptor, as well as a linker to TiO_2.

4. Stability of D131

4.1 Effect of Electrolyte Components on Durability

Basically, electrolytes of DSCs are composed of iodine (I_2), iodide (I^-), amine and organic solvent (nitrile or ester compounds). To elucidate the contribution of each component of the electrolyte on the durability of cells sensitized with D131, the concentration of each solute was changed and durability in the 85°C-dark-test[20] was compared (**Fig. 4**). The efficiency decreased more quickly at higher iodine concentration and higher base concentration. Iodide concentration did not affect the durability of the cells, nor was there a difference between nitrile and ester solvents. In the case of D131,
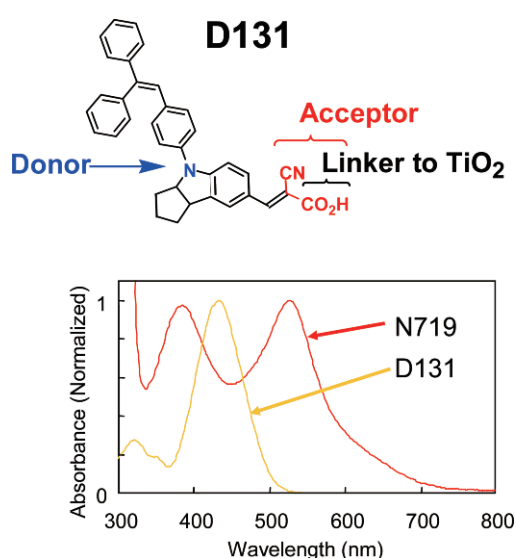

Fig. 3 Structure of D131 and absorption spectrum of D131 in chloroform.

degradation was found to be promoted by amine which is sometimes used as an energy level modifier of TiO_2.

The effect of iodine concentration was investigated in detail (**Figs. 5, 6**). In the 60°C-1 sun-test[20] durability was improved as iodine concentration increased up to 0.2 M (Fig. 5). Efficiency was decreased due to the decrease of J_{sc}, V_{oc} and ff. In contrast, in the 85°C-dark-test, durability decreased as iodine concentration increased (Fig. 6). The efficiency change was mainly due to the decrease of J_{sc}. This result shows that the degradation mechanism is different between the 85°C-dark-test and 60°C-1 sun-test (**Fig. 7**). Dependency on iodine concentration in the 85°C-dark-test suggests

that degradation of dye in the cell is caused by a reaction between the dye and iodine. In the 60°C-1 sun-test, the unstable oxidized dye, which results from electron injection from the S_1 state of the dye into TiO_2, would be reduced after electron injection more quickly as iodine concentration increases. On the other hand, at low iodine concentration, the unstable oxidized dye was reduced more slowly, when the oxidized dye decomposes. For this mechanism, durability would be improved by increasing iodine concentration. Careful optimization of electrolyte composition improved the durability, which is close to N719 in 85°C-dark-test (**Fig. 8**). **Figure 9** shows
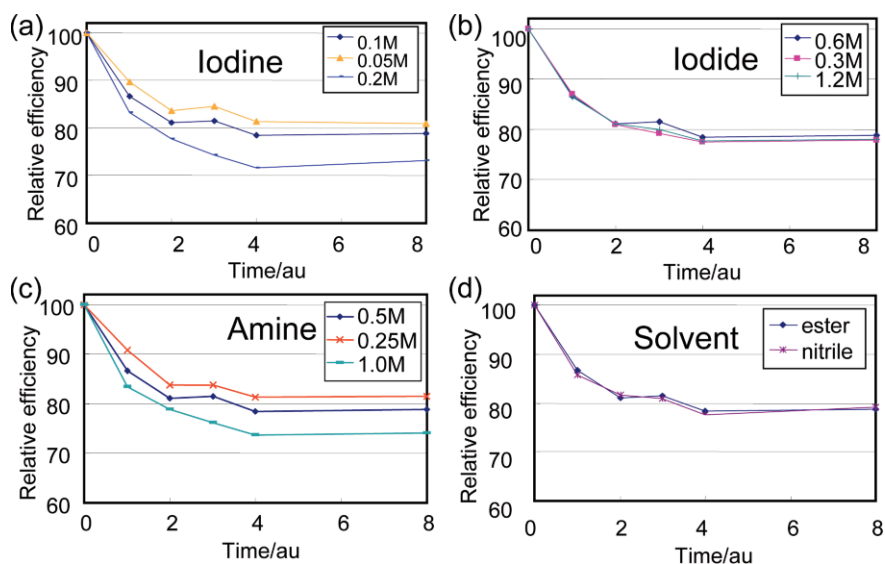

Fig. 4   Dependency of solute (iodine, iodide, amine and solvent) concentration in 85°C-dark test.
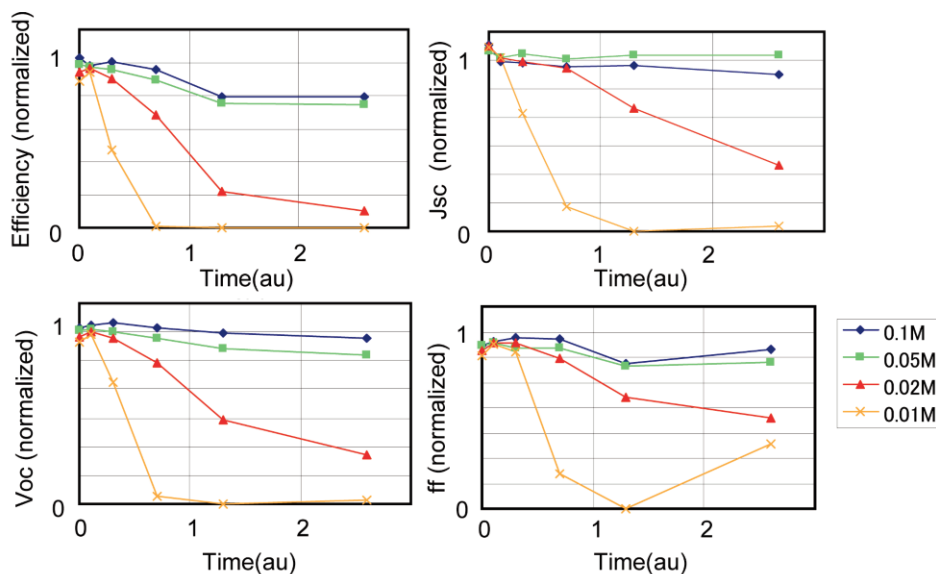

Fig. 5   60°C-1 sun test of D131 cells with electrolyte with various iodine concentrations.

efficiency change in an outdoor test. Thirteen cells were tested and the average, best and worst data of cells were plotted. The efficiency of D131 was stable and decreased gradually over four months. The efficiency change was due to J_{sc} and V_{oc}.

4. 2 Degradation Analysis by Model Reaction

To investigate the structural change of D131 in the cell, a model reaction between dye and electrolyte components in organic solvent was studied. The change of D131 was monitored with an acetonitrile-1-propanol solution of dye (D131) and electrolyte components such as iodine and amine at 85°C in the dark for a few days (**Fig. 10**). Without amine or iodine, no degradation product of D131 was found, but in amine or iodine, a degradation product of D131 was found. After the reaction, the major degradation product (DC131) was separated, purified and identified

with FT-IR, MALDI-TOF MS and ^{1}H-NMR spectroscopies. The FT-IR spectrum shows that the carbonyl peak at 1670 cm^{-1} disappeared in DC131. In the MALDI-TOF MS spectrum, the species at M^{+} = 464.3 corresponds to a mass number of the decarboxylated product of D131 (M^{+} = 508.2) [a decrease of 44 (CO_2) in mass number]. In the ^{1}H-NMR spectrum, an olefinic proton at 5.75 ppm (J = 16.5 Hz) is attributed to a *trans*-phenylacrylnitrile moiety, which is expected to be in decarboxylated D131. According to these spectroscopic data, the major degradation product of these reactions was identified as decarboxylated D131 (DC131, Fig. 10). The structure of DC131 was also confirmed with ^{1}H-^{1}H-COSY (correlation spectroscopy) NMR. It was found that decarboxylation was promoted by iodine or amine as shown in the experiment using model reactions. The rate constants of decarboxylation, k, in iodine or amine solution are shown in **Fig. 11**. Since DC131 has no
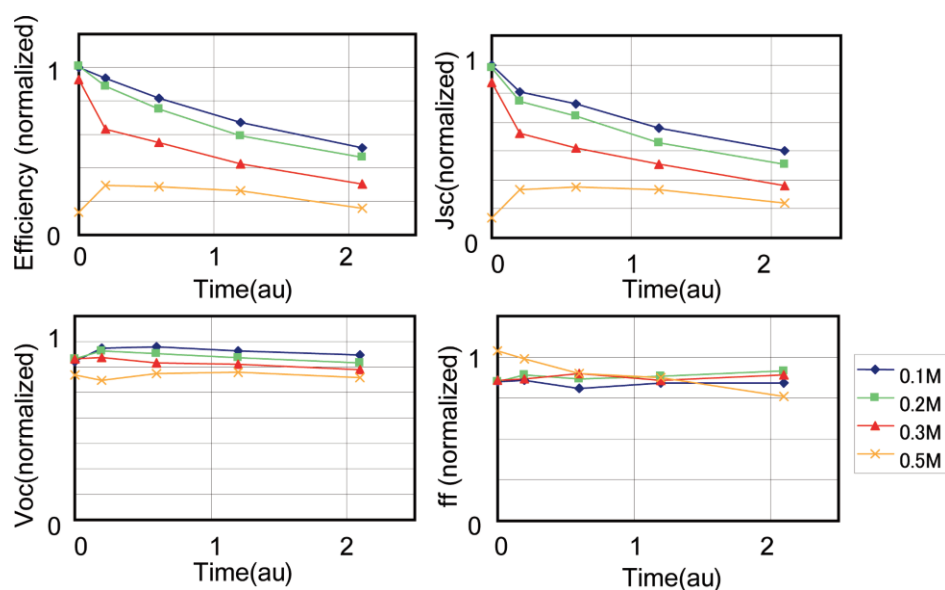

Fig. 6   85°C-dark test of D131 cells with electrolyte with various iodine concentrations.
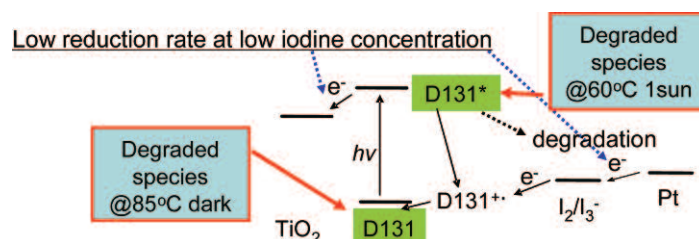

Fig. 7   Degradation mechanisms of D131 cells in 60°C-1 sun test and 85°C-dark test.

carboxylic acid, which is a linker to TiO_2, it can no longer attach to the TiO_2 surface and is liberated in the electrolyte. The loss of dye molecules from the TiO_2 surface is presumed to be one of the degradation mechanisms that lower the performance of the solar cells sensitized with D131.

4. 3 Analysis of Degradation Product in Test Cells

To confirm the degradation mechanism, components in the degraded cells (by 85°C-dark-test and 60°C-1 sun-test) were separated and analyzed (**Fig. 12**). Electrolyte and desorbed species from the photoelectrode surface with base were analyzed with MALDI-TOF MS. DC131 was detected in the photoelectrode and electrolyte (Fig. 12). Meanwhile, in cells stored at r.t. in darkness (designated as initial in Fig. 12), no DC131 was detected in the electrolyte
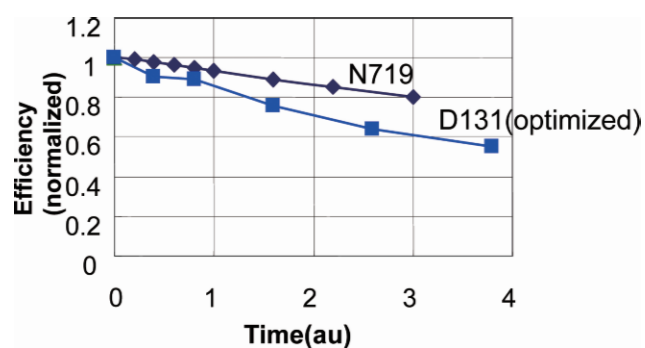
and a small amount of DC131 was detected from the photoelectrode compared to the degraded cell. This result shows that decarboxylation occurs in the test cells during thermal or light soaking durability tests. The decarboxylated product (DC131) which no longer has a linker moiety to TiO_2 dissolves into the electrolyte. Nevertheless, a part of DC131 was found to still remain on the TiO_2 surface.

4. 4 Experimental

Durability Tests

Cell preparation: Photovoltaic cells were constructed as follows. The photoelectrode was prepared by printing TiO_2 and carbon paste on TCO glass, then calcinating at 500°C. The photoelectrode was colorized with 0.5 mM ethanol solution of D131 for 2 days, then washed with ethanol and dried. Electrolytes were prepared with iodine, 1,2-dimethyl-3-propylimidazolium iodide (DMPII), amine and solvent [ester solvent (gamma-butyrolactone) or nitrile solvent (2-methoxypropionitrile)]. Electrolyte was filled between the glass and photoelectrode and sealed with thermoplastic. Finally, the prepared photovoltaic cell was sealed with epoxy resin, and then used for durability tests.

Test conditions: 60°C-1 sun-test was performed by irradiating cells under 1 sun controlling the temperature at 60°C by a load-resistor (10 ohm) connected between electrodes. 85°C-dark-test was performed by heating cells at 85°C in darkness.

Fig. 8   85°C-dark test of D131 cells with optimized electrolyte and N719 cell.

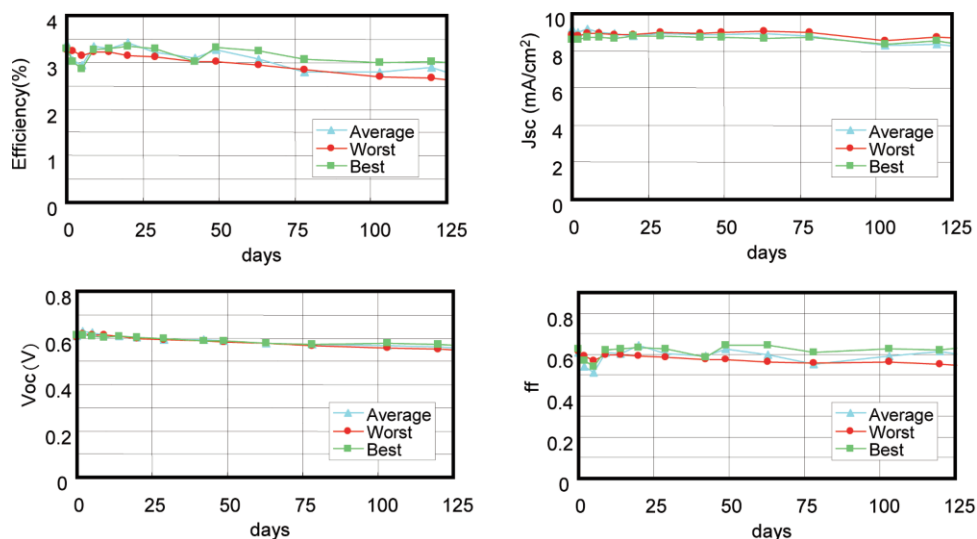**Fig. 9**   Outdoor performance test of D131 cells with optimized electrolyte (average of 10 cells, the worst cell and the best cell).

Performance of the cell was measured at 1 sun at ambient temperature.

Model Reactions

D131 (20 mM) was dissolved in acetonitrile-1-propanol with components of electrolyte such as iodine or amine (40 mM). The solution was heated at 85°C in darkness for a few days. Then, the reaction mixture was evaporated and major reaction products were separated by column chromatography on silica gel eluated with chloroform-hexane.

Analysis of Dye
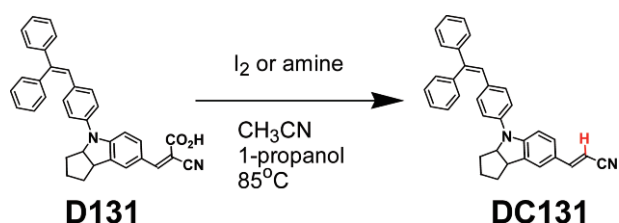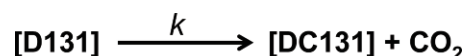
Durability tests of test cells were continued until

Fig. 10 Degradation reaction scheme of D131.

$$[D131] \xrightarrow{k} [DC131] + CO_2$$

$$[DC131] = k\,[D131]$$

condition	K (day^{-1})
D131 20mM, iodine 40mM	0.13
D131 20mM, amine 40mM	0.005

Fig. 11 Decomposition rate of D131 in model reactions.
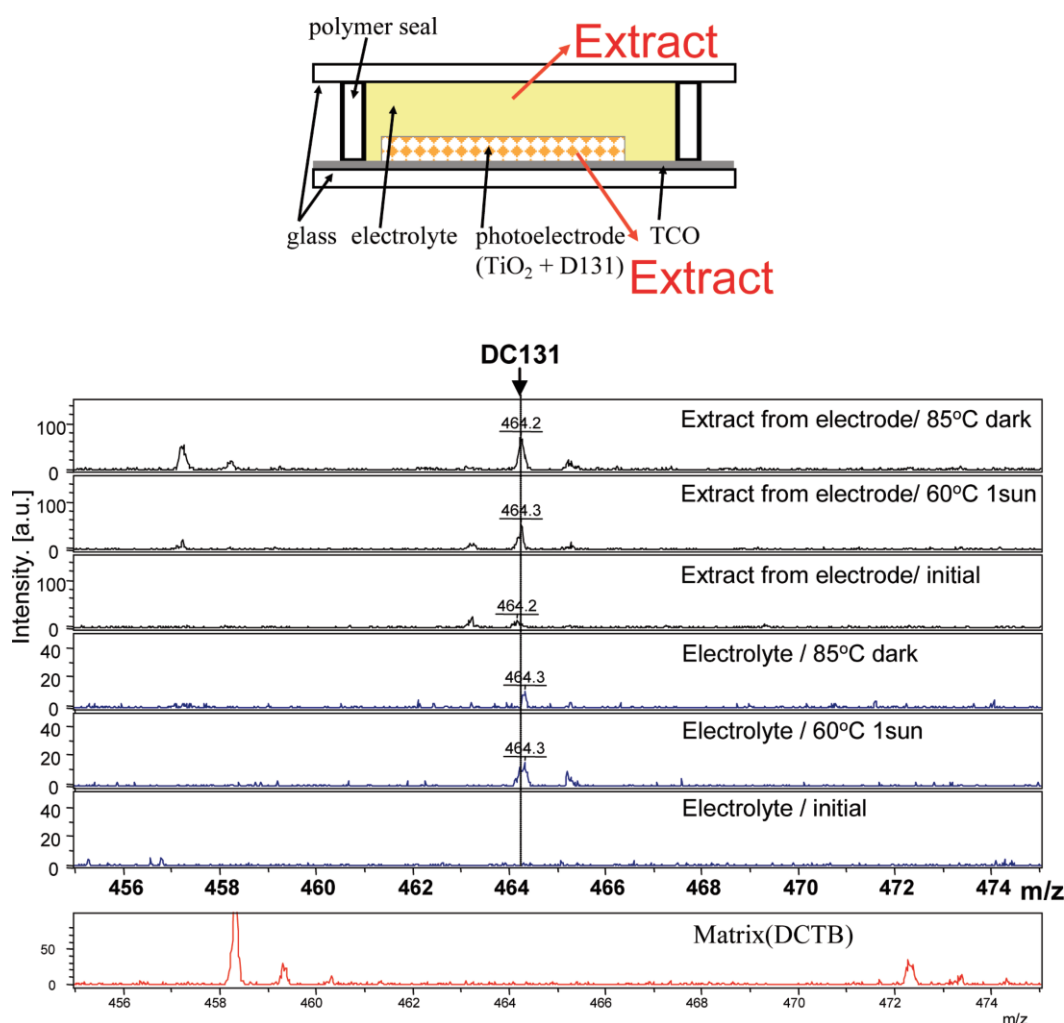

Fig. 12 (a) Cross-section of analyzed cell, (b) MALDI-TOF MS analysis of extracts from electrode and electrolyte of the cells (before and after the 60°C-1 sun test and 85°C-dark test).

their efficiencies reached half of the initial value. The electrolyte was removed from the cells and combined with chloroform extract from cell components. The solution was evaporated and dried under vacuum at 50°C. The photoelectrodes were taken out and washed with acetonitrile and ethanol, and dried. The absorbed dyes were removed from the photoelectrode with acetonitrile solution of triethylamine (5%). The acetonitrile extracts were filtered with filter paper (pore size 200 nm) and evaporated and dried under vacuum, then analyzed with FT-IR (NICOLET MAGNA760), MALDI-TOF MS (matrix-assisted laser desorption/ionization time-of-flight mass spectrometry) (BRUKER DALTONICS ultraflex) and ^{1}H-NMR (JEOL JNM-LA500). FT-IR spectra were recorded with an ATR attachment. DCTB {2-[(2*E*)-3-(4-*tert*-butylphenyl)-2-methylprop-2-enylidene] malononitrile} was used for the MALDI-TOF MS matrix. $CDCl_3$ was used for the NMR solvent. Chemical shifts are given in ppm using TMS as an internal reference.

5. Conclusion

In this article, the chemical stability of sensitizer molecules for dye sensitized solar cells was reviewed. Widely used Ru complex dyes have extremely high turnover (in case of N3 dye, 10^7). For further improvement of the long-term durability of Ru dyes, the decomposition pathway, i.e., the photochemical or chemical decomposition of the NCS group, must be avoided. The interaction between the dye and its surrounding environment such as TiO_2 (hydrophobicity at the surface) and electrolyte (iodide) is a factor determining the durability of the DSC cell. For the DSC sensitized with organic dyes, knowledge of the degradation mechanism, stability and long-term durability is limited. Our study on the stability of a DSC sensitized with a typical donor-acceptor type organic dye (D131) revealed the degradation mechanism of the dye in the DSC cell. The result suggests that in the 85°C-dark condition, degradation of the dye is promoted by iodine and amine in the electrolyte. Under the light irradiation condition, dye decomposition was suggested to start from an unstable oxidized state formed after the electron injection. By model reaction analysis and analysis of decomposition in the cell, the decomposition product of D131 was found to be decarboxylated D131, which has lost the carboxylic acid group that is indispensable for

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Acknowledgements

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Figs. 1, 2
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