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Ionic diffusion in various electrolytes and the implications for dye-sensitized solar cells

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ABSTRACT

The diffusion of I_3^- in volatile and non-volatile electrolytes is investigated to confirm the effects on the performance of dye-sensitized solar cells (DSSCs). The ionic diffusion is controlled by using electrolyte mediums with diverse viscosities including acetonitrile, 3-methoxy-propionitrile, diethylene glycol dimethyl ether, and poly(ethylene glycol dimethyl ether) with three different molecular weights and varying I_3^- concentrations. The diffusion flux in each electrolyte is characterized by scanning electrochemical microscopy. By measuring incident photon to current conversion efficiency, electrochemical impedance spectroscopy, and photovoltaic performance, the optimum diffusion flux of I_3^- is suggested for the best efficiency of DSSCs.

1. Introduction

Since a dye-sensitized solar cell (DSSC) with nanocrystalline metal oxide films was reported, it has been regarded as one of the most promising substitutes for conventional solar cells owing to its low production cost and high energy-conversion efficiency of over 11% [1–3]. The high efficiency, however, can be obtained only when employing volatile electrolytes, which commonly have some restrictions in guaranteeing stability. For the practical application of DSSCs, electrolytes with reliable stability, as well as high efficiency, are highly demanded.

Non-volatile electrolytes including ionic liquids [4–6], oligomers [7,8], and polymers [9,10] have been reported as alternatives to volatile electrolytes. Oligomers, which have been used to investigate non-volatile electrolytes in our group [11–13], have considerable benefits including stability and ionic diffusion properties. Consequently, DSSCs with oligomer electrolytes exhibit long-term and thermal stability as well as high energy-conversion efficiency [7,8,11–13]. In spite of the promising potential of oligomer electrolytes, the DSSCs with oligomer electrolytes, however, have not caught up with those with volatile electrolytes in energy-conversion efficiency. One reason for this inferiority might be a poor understanding of the transport phenomena in the DSSC systems containing non-volatile electrolytes. For example, the chain segmental motions determining the ion transport rate in oligomer and polymer electrolytes [14] and the hopping mechanism to significantly affect ionic diffusion coefficients in highly viscous electrolytes [15] are still under investigation to clarify their influence on the ionic transport in DSSCs. In light of this matter, considerable effort should be devoted to understanding the diffusion behavior in non-volatile electrolytes and finally determining the governing factors of the performance of DSSC employing non-volatile electrolytes.

Ionic diffusion through electrolytes is regarded as a critical factor governing DSSC performance. Diffusion flux largely affects the redox reactions of the photo and counter electrodes of DSSCs. However, defining the effect of the diffusion flux on DSSC performance has still been challenging due to various factors related to diffusion in the electrolytes. In this work, the I_3^- diffusion flux, thought to be the rate-determining factor in the redox reactions, was studied to comprehend the relation between the flux and the DSSC performance. Through the measurements of scanning electrochemical microscopy, the diffusion fluxes in volatile electrolytes with low viscosity including acetonitrile (ACN), 3-methoxy-propionitrile (MPN) and diethylene glycol dimethyl ether (DEGDME) were compared with those in oligomer electrolytes with relatively high viscosity, such as poly(ethylene glycol dimethyl ether) (PEGDME) with three different molecular weights. Each elec-

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trolyte, with varying ionic diffusion fluxes, was applied to DSSCs and the performance was analyzed by measuring incident photon to current conversion efficiency (IPCE), electrochemical impedance spectroscopy (EIS), and photocurrent–voltage to find an optimum ionic flux for the best efficiency of DSSCs.

2. Experimental

2.1. Preparation and characterization of electrolytes

ACN, MPN and DEGDME were selected as volatile electrolyte mediums and oligomers including PEGDME with three different molecular weights of 250, 500, and 1000 g mol^{-1} were utilized as non-volatile electrolytes. 0.8 M of 1-butyl-3-methylimidazolium iodide was added into each electrolyte as an iodide source. The I_3^- concentration in the electrolytes was controlled as 0.01, 0.05, 0.1, and 0.2 M by varying iodine concentration. To determine the I_3^- diffusion flux in each electrolyte, steady-state currents were measured by a scanning electrochemical microscope (SECM, CHI900) at a scan rate of 10 mV s^{-1} [4,11].

2.2. Fabrication of DSSCs

The DSSC preparation has been explained in previous Refs. [11–13]. Transparent fluorine-doped tin oxide (FTO) glass (Pilkington, TEC-8, 8 Ω $sq.^{-1}$) was used as a substrate for the photo and counter electrodes. The substrate was spin-coated with a Ti(IV) bis(ethyl acetoacetato)-diisopropoxide solution (7% w/w in 1-butanol) and annealed at 500 °C for 15 min to construct a TiO_2 blocking layer of the photoelectrode. TiO_2 paste (18NR-T, Dyesol) was cast on the blocking layer using a doctor blade and sintered sequentially at 150 °C for 30 min and 500 °C for 15 min. The TiO_2 layer was about 6.0 μm thick as measured by a surface profiler (KLA-Tencor, Alpha-Step IQ). The TiO_2 electrodes were dipped into purified N719 dye (Solaronix) solution (0.5 mM in absolute ethanol). A Pt layer on the counter electrode was prepared by spin-coating FTO glass with H_2PtCl_6 solution (10 mM in isopropanol) and sintering at 400 °C for 20 min. The sensitized electrode and counter electrode were assembled by a thermal adhesive film of 25 μm thick and filled with various prepared electrolytes. The active area of the sensitized TiO_2 layer was approximately 0.24 cm^2 for all cells, as measured by a digital microscope camera (Moticam 1000).

2.3. Measurement of cell performance

During all measurements, cell temperature was fixed in the range of 25–30 °C using a cooling fan to obtain reliable data of cell performance. The IPCE of DSSCs employing each electrolyte was measured at wavelengths from 300 to 800 nm with a chopping rate of 10 Hz (PV Measurement, Inc.) [16]. A monochromatic beam was generated by a 75 W Xenon lamp and IPCE data were obtained under bias light. Photocurrent–voltage data were collected under 1 sun condition (100 mW cm^{-2}, AM 1.5) calibrated with a Si standard solar cell using a Keithley Model 2400 and a 1000 W Xenon lamp (91193, Oriel). EIS measurements were conducted using a potentiostat (Solartron 1287) and a frequency response analyzer (Solartron 1260). The impedance spectra, with an oscillation level of 10 mV, were examined at open-circuit potential under the same illumination conditions as the measurement of photocurrent–voltage. The obtained data were fitted by using Z-view software. Electron lifetime in the photoelectrode was determined by transient measurements [17,18]. A diode laser (λ = 635 nm) was operated at 3.00 V and stepped down to 2.90 V by a function generator, and the laser intensity was controlled using ND filters.

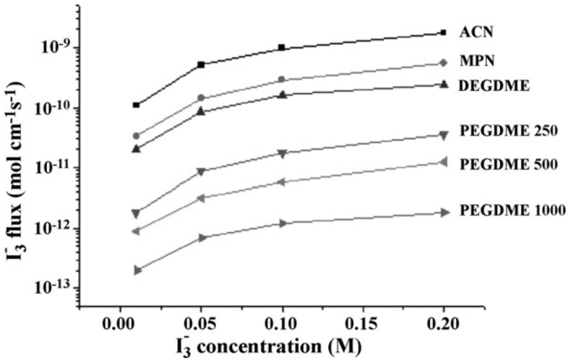

Fig. 1. I_3^- diffusion flux as a function of I_3^- concentration in various electrolytes.

3. Results and discussion

3.1. Diffusion flux of I_3^-

I^-/I_3^- is one of the most common redox couples in DSSC electrolytes owing to its redox potential, reaction rate, diffusion coefficient, and solubility [19–21]. In most cases, I_3^- is a rate-determining component of the ionic diffusion in DSSCs [4,11,12]. Accordingly, the charge transfer of the counter electrode and electron recombination in the photoelectrode are largely affected by I_3^- diffusion [6,22]. Ionic diffusion coefficients have been considered the key factor to determine DSSC performance [23,24]. The diffusion coefficient, however, shows only the diffusion rate of a single molecule, while real diffusion behavior cannot be described perfectly without counting the number of transporting species. Therefore, the total ionic diffusion flux, rather than the diffusion coefficient, may be much more meaningful in estimating the performance of DSSCs. The ionic diffusion flux (DC: D is the ionic diffusion coefficient and C is the bulk concentration of electroactive species) can be evaluated by the following equation:

$$DC = \frac{I_{SS}}{4nFr}$$

where n is the electron number per molecule, F is the Faraday constant, r is the radius of the Pt ultramicroelectrode (5 μm) and I_{SS} is the steady-state current. We assume that the iodine in the electrolyte is totally converted to I_3^-.

I_3^- diffusion flux in various electrolytes is described in Fig. 1. The flux in volatile electrolytes such as ACN, MPN and DEGDME is relatively large (from 10^{-11} to 10^{-8} mol cm^{-1} s^{-1}) compared with that in non-volatile electrolytes (from 10^{-13} to 10^{-10} mol cm^{-1} s^{-1}). In addition, the flux in each electrolyte increases with increasing I_3^- concentration. Thus, a wide range of the diffusion flux can be obtained by using diverse electrolytes and varying I_3^- concentration.

3.2. Incident photon to current conversion efficiency

An optimum ionic flux would allow enough ionic diffusion to conduct redox reactions smoothly, whereas too much flux might bring about side reactions such as recombination between electrons in the photoelectrode and I_3^-. One can estimate whether a diffusion flux is sufficient by investigating the current density (J_{SC}) of cells. With an insufficient amount of diffused ions to reduce oxidized sensitizers and transfer charges of the counter electrode, low J_{SC} would be detected. Since the IPCE value represents the conversion efficiency of incident photon to current, whether the diffusion flux is appropriate can be determined through analyzing the IPCE data.

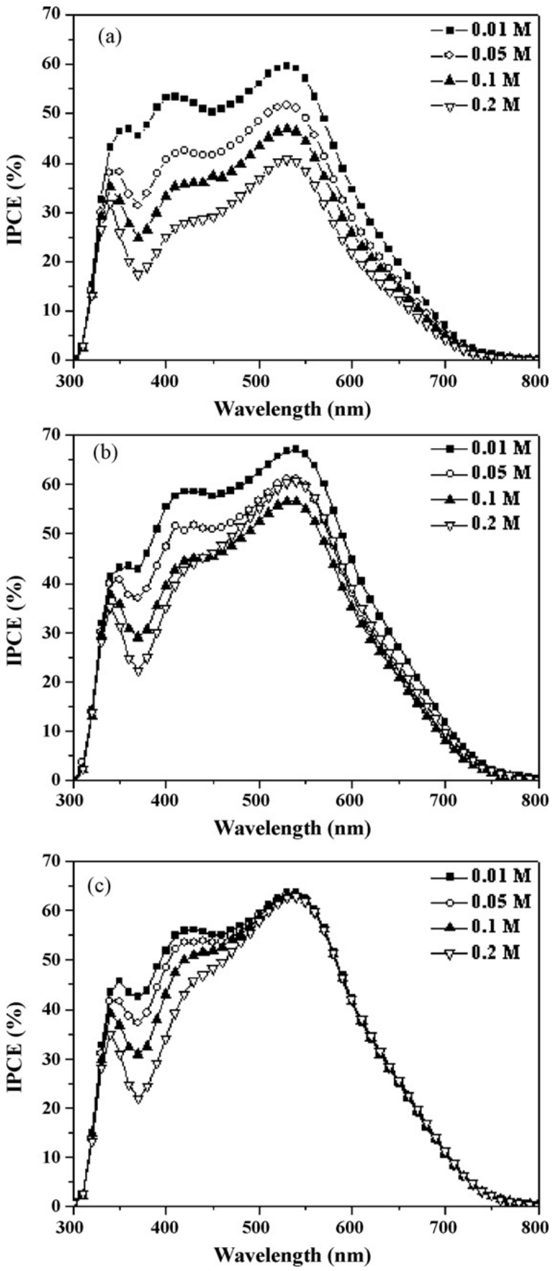

Fig. 2. IPCE of DSSCs employing (a) ACN, (b) MPN, and (c) DEGDME electrolytes.

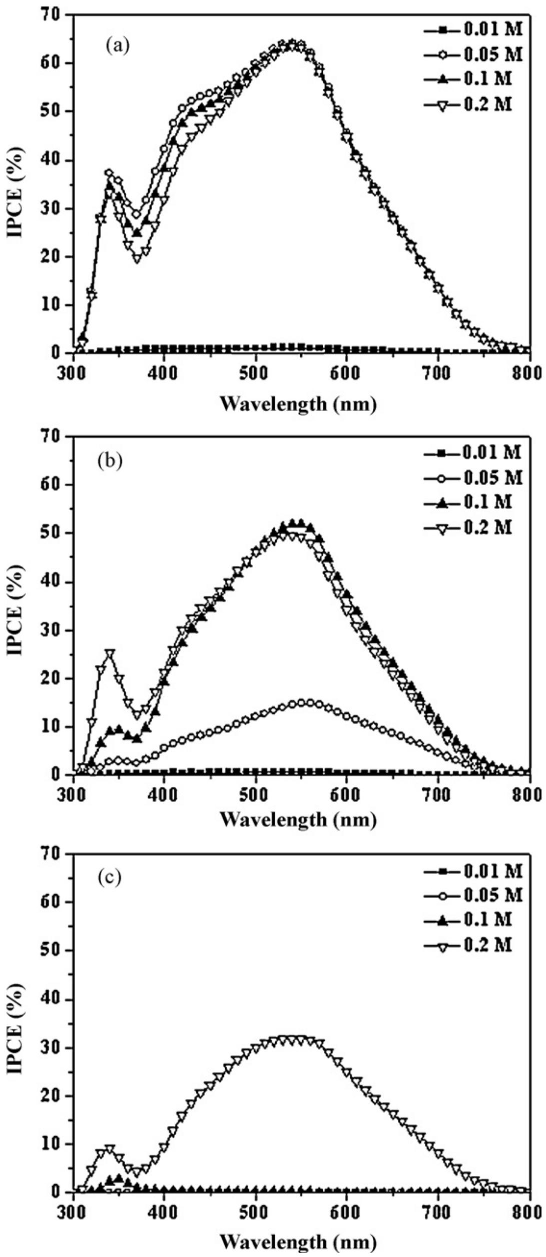

Fig. 3. IPCE of DSSCs employing PEGDME electrolytes with molecular weights of (a) 250, (b) 500, and (c) 1000.

Comparing IPCE data collected from DSSCs employing volatile and non-volatile electrolytes reveals interesting features. The DSSCs employing volatile electrolytes showed the maximum IPCE value at the lowest I_3^- concentration (0.01 M) and the IPCE values decreased with increasing I_3^- concentration (Fig. 2). On the contrary, DSSCs with non-volatile electrolytes had an IPCE value close to zero at the smallest ionic flux (Fig. 3). It implies that DSSCs employing volatile electrolytes have enough ionic flux to conduct redox reactions at the I_3^- concentration as low as 0.01 M while DSSCs with non-volatile electrolytes do not. In addition, it is known that the ionic diffusion coefficient in oligomer electrolytes increases with decreasing molecular weight [11]. Therefore, the PEGDME with the smallest molecular weight (250 g mol^{-1}) resulted in the largest diffusion flux, compared with the other electrolyte compositions containing PEGDME of higher molecular weights at the same I_3^- concentration (Fig. 1). This result is consistent with the IPCE data. DSSCs employing the PEGDME electrolyte with a molecular weight of 250 showed the maximum value at a relatively small diffusion flux (0.05 M of I_3^-) while those employing PEGDME of 1000 g mol^{-1} required a higher concentration of I_3^- to maximize the IPCE value. The proportional tendency between the diffusion flux and the IPCE value is conspicuous when employing PEGDME of 500 g mol^{-1} (Fig. 3(b)).

In DSSCs with volatile electrolytes, IPCE values decrease with increasing ionic flux. The much lower viscosity of volatile electrolytes facilitates ionic transfer, providing enough ionic flux to conduct redox reactions even at an I_3^- concentration as low as 0.01 M. The excess diffused ions can participate in the recombination between injected electrons in TiO_2 and I_3^- and thus cause the reduced IPCE value.

3.3. Electrochemical impedance spectroscopy

EIS measurements have been frequently utilized to investigate charge transfer resistances at the interfaces that largely affect DSSC

Table 1
Charge transfer resistance of the counter electrode in DSSCs employing various electrolytes.

Electrolyte	I_3^- concentration			
	0.01 M	0.05 M	0.1 M	0.2 M
	Charge transfer resistance (Ω)			
ACN	4.66	4.43	3.53	4.67
MPN	7.18	5.66	4.36	4.02
DEGDME	22.62	11.30	6.75	5.38
PEGDME 250	102.41	13.81	9.84	6.77
PEGDME 500	213.91	23.36	16.07	13.94
PEGDME 1000	386.82	56.01	16.86	14.16

performance [14,25,26]. In this study, charge transfer of the counter electrode (the interface between the Pt layer and the electrolyte) was measured to understand the relation between diffusion flux and the charge transfer resistance. Table 1 illustrates the charge transfer resistances of the counter electrode, demonstrating that the charge transfer resistance decreased with increasing I_3^- flux. In other words, increasing I_3^- concentration in the same electrolyte medium resulted in the reduction of the charge transfer resistance. Also, DSSCs employing volatile electrolytes exhibit a much smaller charge transfer resistance than those with non-volatile electrolytes at the same I_3^- concentration, implying that the charge transfer reaction between the Pt layer and the electrolyte was smoothly conducted, owing to the sufficient ionic flux. In DSSCs with ACN- and MPN-based electrolytes, which have low viscosity and large ionic diffusion flux, the charge transfer resistance was very small even at an I_3^- concentration as low as 0.01 M. On the contrary, DSSCs employing other electrolytes required a relatively large ionic flux (higher than 0.1 M of I_3^-) to considerably suppress the charge transfer resistance. The small charge transfer resistance of the counter electrode would be helpful to enhance the open-circuit voltage (V_{OC}) and the fill factor (FF) of DSSCs.

DSSCs have another charge transfer of the photoelectrode (the interface between the TiO_2 nanocrystalline electrode and the electrolyte). The charge transfer resistance is mainly affected by the interfacial contact between the TiO_2 electrode and electrolyte [14,25]. DSSCs with non-volatile electrolytes showed larger charge transfer resistances of the photoelectrode than those with volatile electrolytes (Table S1 in the supplementary data). The larger resistance might result from the large viscosity and poor permeability of non-volatile electrolytes. Even though the charge transfer resistance of the photoelectrode can also affect DSSC performance, this study does not address that issue, because it is not directly related to the diffusion flux [7,27].

3.4. Electron lifetime

Electron lifetime in the TiO_2 electrode of DSSCs was characterized by transient measurements, and the data were fit using an exponential function [17,18]. Most DSSCs employing volatile electrolytes showed similar values of electron lifetime, as did non-volatile electrolytes. Hence, data from DSSCs employing MPN and PEGDME 500 electrolytes were chosen to scrutinize electron lifetime behavior against Jsc on behalf of volatile and non-volatile electrolytes, respectively (Fig. 4). In previous literature [11,12], it was reported that the electron lifetime decreases with an increasing I_3^- concentration since the large I_3^- diffusion flux induces electrons in the TiO_2 electrode to recombine with I_3^-. This study confirms those results. When comparing the DSSCs with MPN and PEGDME 500 electrolytes, the latter was found to have a much longer electron lifetime due to the smaller I_3^- diffusion flux in non-volatile electrolytes. In other words, when the ionic diffusion flux is small, the collision frequency between electrons in the TiO_2 electrode and I_3^- decreases, resulting in a drop of recombination and an increase of electron lifetime [21,28]. The slow recombination rate prevents the down of the Fermi level of the TiO_2 electrode and thereby the Voc relatively increases [29,30].

3.5. Photovoltaic performance

The photovoltaic characteristics of DSSCs employing volatile and non-volatile electrolytes as a function of diffusion flux are described in Figs. 5 and 6, respectively. (The detailed data are presented in Table S2 and S3 in the supplementary data.) In order to construct a firm reliability in measuring photovoltaic performance, at least five cells for each electrolyte system were tested. Photovoltaic performance was largely affected by the diffusion flux of I_3^-. The V_{OC} of all cells decreased with increasing I_3^- flux. A large I_3^- diffusion flux decreases the electron lifetime due to recombination between the electrons and I_3^- and thereby shifts the Fermi level of the TiO_2 electrode downward. Since V_{OC} is the potential difference between the Fermi level of the TiO_2 electrode and the redox potential of the electrolyte, the smaller V_{OC} was detected with increasing the diffusion flux of I_3^-. On the contrary, the FF of DSSCs increased with increasing the I_3^- concentration; a larger I_3^- diffusion flux resulted in a larger FF value. The inverse proportional relationship between the FF value and charge transfer resistance was confirmed in DSSCs employing non-volatile electrolytes with large charge transfer resistance and a small FF (Fig. 6).

The most prominent difference between DSSCs with volatile and non-volatile electrolytes was found in the J_{SC} change in terms of the I_3^- flux. The J_{SC} of DSSCs employing volatile electrolytes decreased with increasing I_3^- flux, while those with non-volatile electrolytes showed the opposite trend. When the concentration

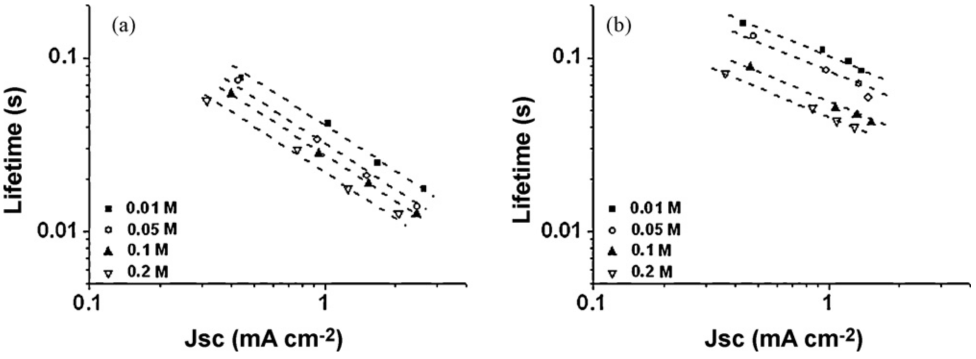

Fig. 4. Electron lifetime in TiO_2 electrode of DSSCs with (a) MPN and (b) PEGDME 500 electrolytes.

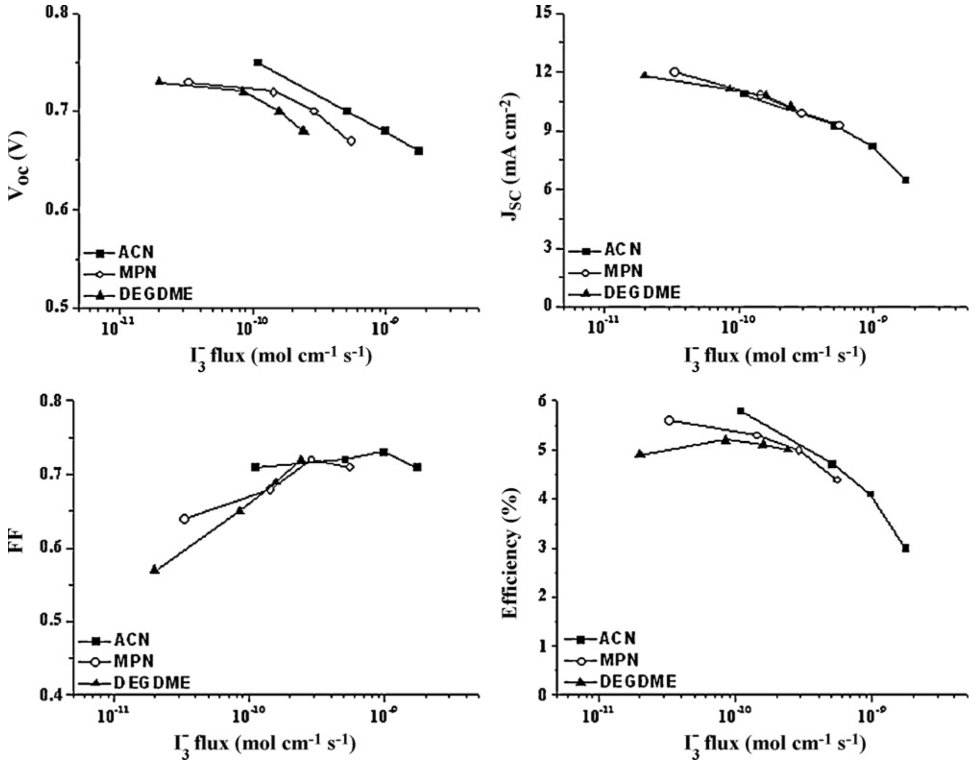

Fig. 5. Photovoltaic characteristics of DSSCs employing volatile electrolytes as a function of I_3^- diffusion flux.

of I_3^- is low, non-volatile electrolytes do not have sufficient I_3^- flux to properly conduct redox reactions. Therefore, increased I_3^- flux in non-volatile electrolytes allows the facilitation of the redox reaction and thereby improving J_{SC}. In volatile electrolytes, however, recombination between electrons in the TiO_2 electrode and excess I_3^- ions is likely to reduce the J_{SC} when the ionic flux increases. This opposite J_{SC} trend between volatile and non-volatile electrolytes made the efficiency of the DSSCs quite different. The efficiency of the DSSCs with non-volatile electrolytes increased with increasing I_3^- diffusion flux, whereas those employing volatile

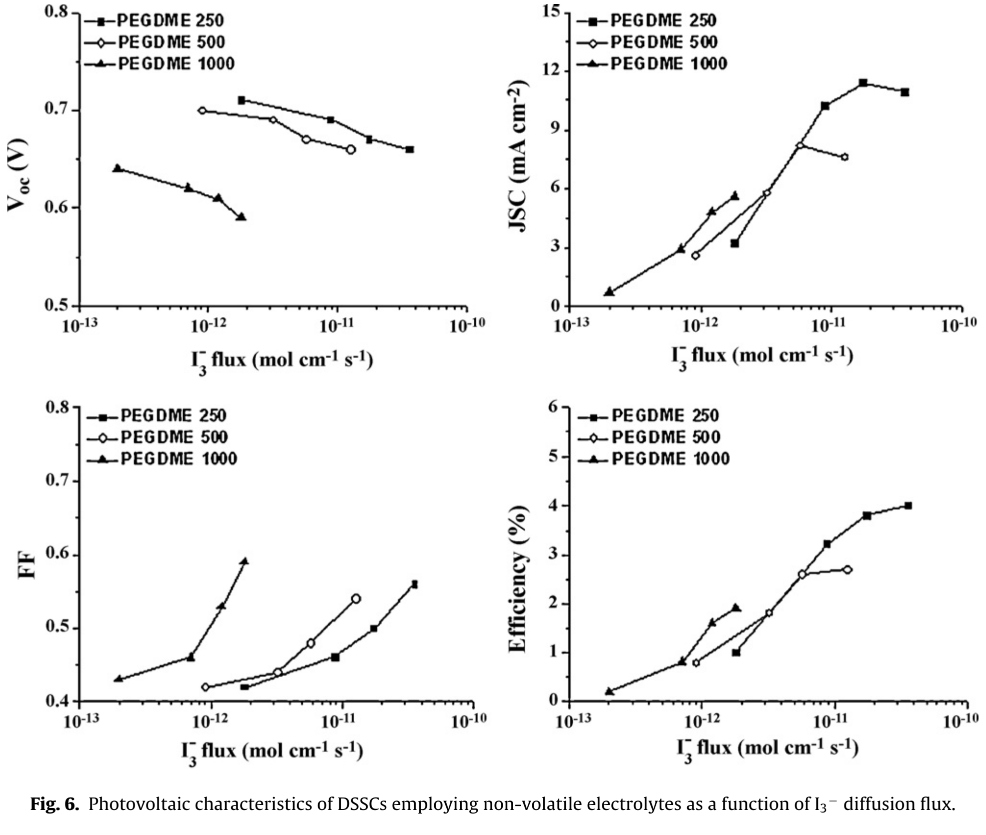

Fig. 6. Photovoltaic characteristics of DSSCs employing non-volatile electrolytes as a function of I_3^- diffusion flux.

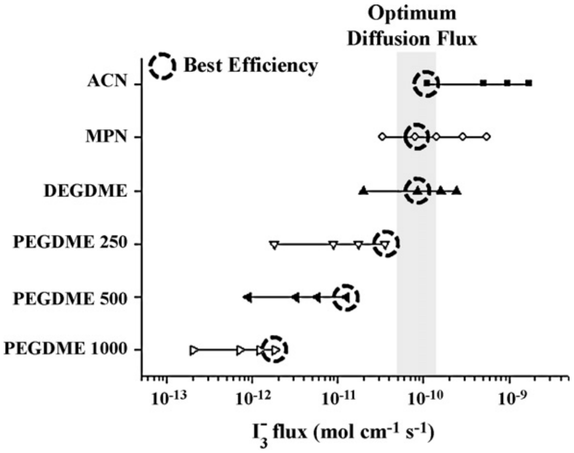

Fig. 7. Optimum $I_3{}^-$ diffusion flux for cell efficiency.

electrolytes showed optimal efficiency at a relatively small diffusion flux (0.3–2 × 10^{-10} mol cm^{-1} s^{-1}).

3.6. Optimum diffusion flux

Analyzing the photovoltaic performance (Figs. 5 and 6) suggests an optimum $I_3{}^-$ diffusion flux for the best energy-conversion efficiency. To clearly specify the optimum flux, MPN electrolyte containing 0.03 M of $I_3{}^-$ with a diffusion flux of 0.81 × 10^{-10} mol cm^{-1} s^{-1} was further investigated. In comparing the efficiency of DSSCs employing MPN electrolytes containing 0.01, 0.03 and 0.05 M of $I_3{}^-$, DSSCs using MPN electrolytes with 0.03 M of $I_3{}^-$ showed the best energy-conversion efficiency (5.7%). Fig. 7 describes the diffusion flux producing the best efficiency with each electrolyte. Considering the DSSC efficiencies, the optimum $I_3{}^-$ diffusion flux is about 10^{-10} mol cm^{-1} s^{-1} regardless of the kind of electrolytes and each electrolyte should have different $I_3{}^-$ concentration to achieve the optimum flux. Based on this result, even the non-volatile electrolyte with the largest diffusion flux (PEGDME with a molecular weight of 250) still requires a larger diffusion flux to enhance efficiency. Hence, a strategy to raise the diffusion flux in non-volatile electrolytes is highly essential to obtain higher efficiency. The increased concentration of $I_3{}^-$ to improve the diffusion flux, however, is not desirable, since the high concentration results in light absorption by $I_3{}^-$ causing a decrease of the available light for the sensitizer [6]. One promising solution might be to introduce inorganic nanoparticles into non-volatile electrolytes, allowing ions to propagate well with high diffusion flux in non-volatile electrolytes, as well as donating mechanical stability to the solar cells [4,6,8].

4. Conclusions

This study investigated the relation between $I_3{}^-$ diffusion flux in electrolytes and DSSC performance. The ionic diffusion flux significantly affects redox reactions of the photoelectrode as well as charge transfer of the counter electrode and, consequently, DSSC performance. By characterizing DSSCs with various electrolytes, the optimum diffusion flux of $I_3{}^-$ (10^{-10} mol cm^{-1} s^{-1}) producing the best efficiency and $I_3{}^-$ concentration in each electrolyte to possess the optimum flux are suggested. Based on these results, it is confirmed that the smaller diffusion flux of $I_3{}^-$ in non-volatile electrolytes compared to volatile electrolytes is a critical factor in restricting the efficiency of DSSCs employing non-volatile electrolytes.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jphotochem.2010.03.010.

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