

# A New Ionic Liquid for a Redox Electrolyte of Dye-Sensitized Solar Cells

Man Gu Kang, Kwang Sun Ryu, Soon Ho Chang, and Nam-Gyu Park

A new ionic liquid, 1-vinyl-3-heptylimidazolium iodide (VHpII), was synthesized and applied as a redox electrolyte for dye-sensitized solar cells. The chemical structure of the synthesized VHpII was confirmed using  $^1\text{H}$  NMR. Thermogravimetric analysis showed that the VHpII was stable for thermal stress of up to  $250^\circ\text{C}$ . The energy conversion efficiencies of the VHpII-based dye-sensitized solar cells were investigated in terms of the effect of a lithium iodide addition. A solar cell containing the redox couple of VHpII and iodine showed a conversion efficiency of 2.63% under 1 sun light intensity at AM 1.5. Adding 0.4 M LiI results in a conversion efficiency of 3.63%, which was an improvement of about 40%. The increased conversion efficiency was ascribed to an increase in external quantum efficiency.

**Keywords:** Dye-sensitized solar cell, ionic liquid, 1-vinyl-3-heptylimidazolium iodide, molten salt, redox electrolyte.

## I. Introduction

The solar cell, which converts light into electrical energy through dye-sensitized nanocrystalline  $\text{TiO}_2$  films, was developed by Grätzel et al [1]. Since then, dye-sensitized solar cells (DSSCs) have been investigated extensively as a low-cost alternative to conventional inorganic *pn* junction-type solar cells. When DSSC is illuminated, the dye, excited by a photon, injects an electron into the conduction band of  $\text{TiO}_2$ . The oxidized dye is subsequently regenerated by a redox electrolyte. The redox electrolyte is generally composed of an  $\text{I}_3^-/\text{T}^-$  redox couple in an organic solvent such as acetonitrile or 3-methoxypropionitrile [1]. Such organic solvents, however, may cause leakage due to an increase in temperature during illumination, which has a bad influence on long-term stability. To address the issue with organic solvent-based redox electrolytes, several attempts have been made to replace them with p-type inorganic materials [2], [3], organic hole conductors [4]–[7], or solid polymer electrolytes [8]–[13]. Although the nonliquid-based electrolytes seem to solve the problem of leakage, they showed lower conversion efficiencies than those achievable with the organic liquid-based electrolytes. A relatively low conversion efficiency is associated with poor contact between dye-adsorbed nanoparticles and solid hole conductors or polymers within mesoporous film. Ionic liquids (often referred to as molten salt) might be better alternatives because a high photovoltaic performance as well as physical stability is expected due to ionic liquids being more likely to come into contact with dye-on-nanoparticles.

Since the discovery of the first ambient temperature ionic liquid in 1951 [14], many classes of molten salts have been investigated. Imidazolium-based air- and water-stable ionic liquids were reported by Wilkes and Zaworotko [15], and hydrophobic and highly conductive molten salts were

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investigated by Bonhote et al [16]. In recent years, room temperature molten salts have been applied as both iodide sources and solvents for redox electrolytes in DSSCs [17], [18]. Both 1-methyl-3-alkylimidazolium iodide and 1,2-dimethyl-3-alkylimidazolium iodide are mostly used for DSSCs.

We have designed an air-stable molten salt containing a vinyl group instead of a methyl or ethyl group at the C(1) position of the imidazole ring. Since a vinyl substituent is expected to offer a polymerization reaction, a vinyl-contained imidazolium iodide can be a good monomer to make a polymer electrolyte-contained poly iodide. In this paper, we report on the synthesis and characterization of a new molten salt, 1-vinyl-3-heptylimidazolium iodide (VHpII). The photovoltaic performance of VHpII-contained dye-sensitized solar cells is investigated. We also look into the effects of a lithium iodide addition into ionic liquid VHpII on conversion efficiencies.

## II. Experiment

We synthesized 1-vinyl-3-heptylimidazolium iodide as follows. A 20 mL amount of 1-vinylimidazole (Aldrich, 99+%) and 200 mL of trichloroethylene (Aldrich, 99+%) were placed in a three-neck flask, to which a slight excess of heptyl iodide (40 mL) was dripped at a rate of approximately one drop per second under an Ar atmosphere. The solution was heated for three hours at 70°C under vigorous stirring. Two liquid phases formed during this process. The product was collected by the difference of polarity using a separatory funnel and washed with trichloroethylene to remove the unreacted reagents in the product [19]. The synthesized products were confirmed by <sup>1</sup>H NMR spectroscopy (300 MHz Bruker DRX300), using CD<sub>3</sub>OD as a solvent. The thermogravimetric property of the synthesized VHpII was investigated using an SDT 2960 simultaneous DSC-TGA (TA Instrument) at a heating rate of 5°C/min in air.

Anatase TiO<sub>2</sub> colloids were prepared by the hydrothermal reaction of titanium isopropoxide (Aldrich, 97%) at 200°C for 12 h in an autoclave apparatus (Parr, 600 mL). The autoclaved colloidal solution was condensed to a TiO<sub>2</sub> concentration of 12 wt%. A viscous slurry was prepared by adding 20 wt% polyethylene glycol (Fluka, MW 20,000) and 20 wt% polyethylene oxide (Aldrich, MW 100,000) to the colloid solution with respect to the amount of TiO<sub>2</sub>. The slurry was coated on a SnO<sub>2</sub>:F conducting glass (Pilkington, TEC 8) using the doctor blade technique and annealed at 500°C for 30 min in air. The annealed film was estimated to be about 9 μm as measured by a Tencor alpha-step 500 profiler.

The TiO<sub>2</sub> electrodes were immersed in 3×10<sup>-4</sup> M Ru(II)LL'(NCS)<sub>2</sub> (L=2,2'-bipyridyl-4,4'-dicarboxylic acid, L'= L=2,2'-bipyridyl-4,4'-ditetrabutylammonium carboxylate,

Solaronix) ethanol solution for over 24 h at room temperature. Transparent Pt counter electrodes were prepared by spreading a few drops of a 5 mM hydrogen hexachloroplatinate(IV) hydrate (Aldrich, 99.9%) 2-propanol solution on SnO<sub>2</sub>:F conducting glasses, followed by heating at 450°C for 30 min. The dye-coated TiO<sub>2</sub> electrode and the Pt-coated counter electrode were sandwiched using sealing materials (Surlyn 1702, Dupont) under a pressure of 200 kPa/cm<sup>2</sup> at about 100°C.

The redox electrolytes were prepared by dissolving iodine in VHpII with the mole ratio of [VHpII]/[I<sub>2</sub>] = 20, where 4-tert-butylpyridine (0.125 M) and lithium iodide (0, 0.2, 0.4 and 0.6 M) were added. The electrolytes were introduced into the cell through one of the two small holes drilled in the counter electrode by capillary action. The holes were then covered and sealed with small squares of Surlyn and microscope objective glass. The active area of the cell was about 0.25 cm<sup>2</sup>.

The photocurrent (*J*)-voltage(*V*) curve was measured with a Keithley 2400 source meter. A 1000 W Xenon lamp (Oriel, 91193) served as a light source. The light intensity was adjusted using a reference Si cell (Fraunhofer Institute for Solar Energy System) for approximating 1 sun light intensity of 100 mW/cm<sup>2</sup>. The incident photon-to-current conversion efficiency (IPCE) was measured at the low chopping speed of 5 Hz using an IPCE system (PV Measurement Inc.) equipped with a halogen source and a broad band bias light for approximating 100 mW/cm<sup>2</sup>. The IPCE system was calibrated using a silicon reference photodiode (G587, PV Measurement Inc.).

## III. Results and Discussion

Figure 1 shows the <sup>1</sup>H NMR spectrum of the synthesized VHpII, where all the peaks are indexed. The peaks appearing in the range from 0 to 5 ppm are assigned to protons at the heptyl group. In particular, the peaks at 4.4 to 4.45 ppm are assigned to methylene (CH<sub>2</sub>) protons, which are linked to the N in the imidazolium ring.

The peaks appearing in the range of 5.46 to 6.12 and 7.39 to 7.48 ppm correspond to C=CH<sub>2</sub> and CH=C protons of the vinyl group, respectively. The protons within the ring are confirmed from the peaks at 7.97 to 8.19 ppm.

Figure 2 shows the thermogravimetric analysis (TGA) curves of VHpII. No weight loss is observed up to 250°C. Moreover, a five-day semi-long-term test at 150°C shows a little weight loss of about 3%, indicating that the synthesized VHpII is thermally stable.

Figure 3 shows the *J*-*V* curves of DSSC-contained VHpII ionic liquids and the effect of an LiI addition. Short-circuit photocurrent density (*J*<sub>sc</sub>), open-circuit voltage (*V*<sub>oc</sub>), fill factor (*FF*), and conversion efficiency (*η*) are summarized in Table 1.

Dye-sensitized solar cells containing VHpII and iodine

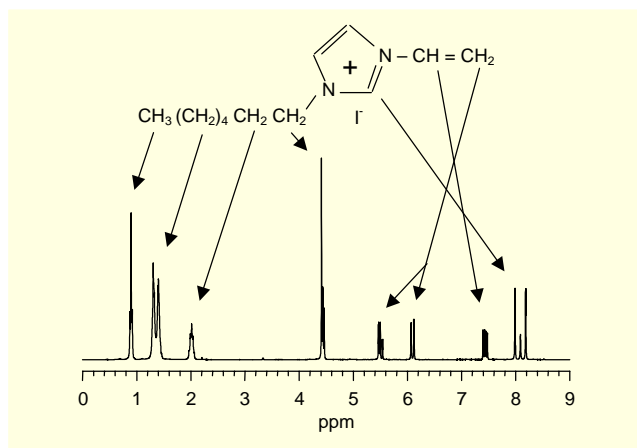


Fig. 1.  $^1\text{H}$  NMR spectrum of the synthesized VHpII.

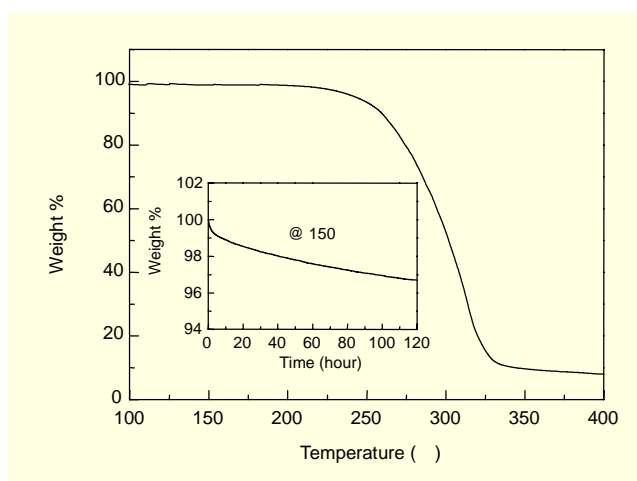


Fig. 2. The TGA curve of VHpII measured at a heating rate of  $5^\circ\text{C}/\text{min}$  in air. The inset shows a plot of weight loss of VHpII at  $150^\circ\text{C}$  as a function of time.

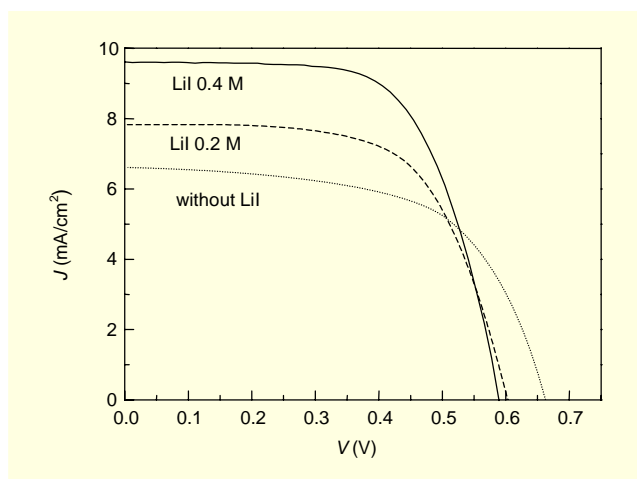


Fig. 3.  $J$ - $V$  curves of dye-sensitized solar cells containing VHpII ionic liquids with and without LiI at AM 1.5 ( $100\text{ mW}/\text{cm}^2$ ).

exhibit an overall conversion efficiency of 2.63%. When LiI is added, the photovoltaic performance is improved. The photocurrent increases and the voltage decreases with an increasing LiI concentration. Consequently, a maximum efficiency of 3.63% is obtained in this experiment with 0.4 M LiI in a VHpII molten salt electrolyte. Although a slight increase in the photocurrent is observed upon the addition of a LiI of more than 0.4 M, the conversion efficiency is no longer improved due to decreases in both voltage and fill factor. The conversion efficiency of a VHpII-based DSSC seems to be not as good as an organic liquid-based one; however, the former is more beneficial in a practical point of view because of thermal stability. In addition, the absolute efficiency value is expected to improve further in cases where the structure and thickness of  $\text{TiO}_2$  films are optimized.

Table 1. The effect of a LiI addition to VHpII on the photovoltaic characteristics of dye-sensitized solar cells.

Electrolyte <sup>a)</sup>	$V_{oc}$ (V)	$J_{sc}$ ( $\text{mA}/\text{cm}^2$ )	$FF$	$\eta$ (%)
VHpII	0.66	6.63	0.60	2.63
VHpII LiI 0.2 M	0.60	7.88	0.63	2.98
VHpII LiI 0.4 M	0.59	9.61	0.64	3.63
VHpII LiI 0.6 M	0.56	10.51	0.61	3.59

a) molar ratio of  $[\text{VHpII}]/[\text{I}_2] = 20$

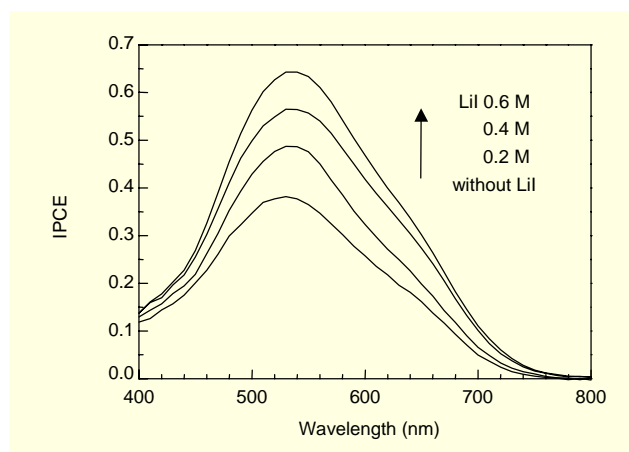


Fig. 4. IPCE spectra of VHpII-contained dye-sensitized solar cells as a function of LiI concentration.

Figure 4 shows the effect of LiI concentration in a VHpII molten salt electrolyte on IPCE as a function of wavelength. The maximum IPCE occurs at around 530 nm, with the highest IPCE being 0.64 for a 0.6 M LiI. The IPCE enhancement with an increasing LiI concentration is greatly consistent with the result of an increase in the photocurrent, as

shown in Fig. 3. The increased  $J_{sc}$  by the addition of LiI is therefore as a consequence of the increased external quantum efficiency.

Regarding the decreased  $V_{oc}$  by LiI addition, we consider first the change in redox potential due to increased iodide concentration. For the case of a 0.4 M LiI addition, the potential increase is estimated based on (1),

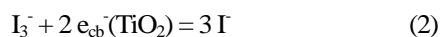
$$E = E^0 + (0.026/n)\ln([I^-]^3/[I_3^-]), \quad (1)$$

where  $n$  is the mole number of electrons and  $E^0$  is the formal potential. Iodide concentration is calculated as follows. The mole ratio of electrolytes is VHpII :  $I_2$  : LiI = 150 : 7.5 : 10 $x$  ( $x$  = LiI concentration). Iodide concentration  $[I^-]$  is 142.5 and 146.5 for  $x = 0$  and  $x = 0.4$ , respectively, assuming that  $I_2$  is completely converted to  $I_3^-$  by reaction with  $I^-$ . The calculated potential of  $E^0 + 0.167$  V is increased to  $E^0 + 0.168$  V upon adding 0.4 M LiI. Therefore, a redox potential change by only 1 mV cannot explain the observed voltage difference of 70 mV. A more plausible explanation is related to the band edge movement.

When comparing the maximum IPCE of VHpII with and without LiI, a slight red shift is observed for the LiI-contained VHpII electrolyte. The shift to a higher wavelength is due to a small cation effect, associated with a decrease in  $V_{oc}$ . A similar observation was reported in the comparative study of LiI and 1,2-dimethyl-3-hexyl imidazolium iodide [20]. The addition of a small  $Li^+$  cation in VHpII can cause the band edges to shift to a more positive potential because the  $Li^+$  cations adsorbed on the  $TiO_2$  surface are likely to induce the potential drop across the Helmholtz layer. As a result,  $V_{oc}$  is expected to decrease.

The increase in  $J_{sc}$  can also be related to conduction band edge movement. The positive shift of the  $TiO_2$  conduction band by Li cations allows low lying excited states of the adsorbed dye to inject electrons, resulting in an enhanced photocurrent.

Figure 5 compares the current-voltages under illumination and in the dark for the dye-sensitized solar cells with and without 0.4 M LiI in VHpII. It is noted that the addition of LiI into a VHpII electrolyte results in an increase in the dark current, implying an increase in the recombination rate. The conduction band electrons can be recombined with  $I_3^-$  ions at the interface of the  $TiO_2$  surface and electrolyte, which can be seen in (2).



A large dark current at a given forward bias voltage underlines a large loss of electrons in the  $TiO_2$  conduction band. The increased back electron transfer is attributable to less

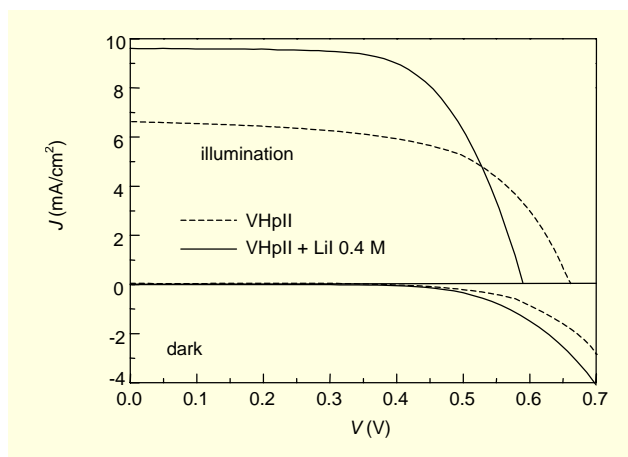


Fig. 5. Comparison of current-voltages under illumination and in the dark for DSSCs with and without LiI in VHpII.

screening of the conduction band electrons from  $I_3^-$  ions in the presence of  $Li^+$  ions adsorbed on the  $TiO_2$ /electrolyte interface, compared with that caused by more hydrophobic VHpI $^+$  cations. The reduction reaction (2) in the presence of LiI is therefore enhanced, leading to an increase in the back electron transfer. The increase in the  $I_3^-$  reduction rate tends to decrease  $V_{oc}$  based on the nonideal diode equation in [21],

$$V_{oc} = (kT/e) \ln(I_{inj}/n_{cb}k_{et}[I_3^-]), \quad (3)$$

where  $I_{inj}$  is the charge flux from the sensitized injection,  $n_{cb}$  is the surface electron concentration at the  $TiO_2$  surface, and  $k_{et}$  is the rate constant of the  $I_3^-$  reduction.

The fill factor is found to increase slightly with the addition of LiI, as shown in Table 1, suggesting a series resistance decrease, as evident by the increased slope of the  $J$ - $V$  curves around  $V_{oc}$  of the LiI-added cell in Fig. 5. This implies that the increase of the fill factor can be ascribed to an increase of the conductivities of the electrolytes in the presence of LiI, as compared with those without LiI in VHpII.

Finally, we investigate the dependence of the photocurrent and voltage on light intensity for a DSSC with 0.4 M LiI. The photocurrent shows a linear relationship with the light intensity, which means little mass transport of the redox shuttle. With the increase of the light intensity, however, the  $V_{oc}$  increases logarithmically in accordance with the kinetic analysis, as shown in (3) [22].

## IV. Conclusion

We synthesized an imidazolium iodide-based molten salt-contained vinyl group. Synthesized 1-vinyl-3-heptyl imidazolium iodide was found to be stable for thermal stress up to 250°C and non volatile at 150°C. The newly prepared

molten salt was applied as an electrolyte for a dye-sensitized solar cell. Although the synthesized molten salt shows a lower conversion efficiency than organic liquid-based dye-sensitized solar cells, it is better than the reported ionic liquids in the absence of an organic solvent. Since the vinyl group in the molten salt can offer polymerization, our synthesized molten salt is useful for solid-state dye-sensitized solar cells. Polymerization of 1-vinyl-3-heptyl imidazolium iodide and its application to dye-sensitized solar cell are under study.

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