Polymer-Doped Molten Salt Mixtures as a New Concept for Electrolyte Systems in Dye-Sensitized Solar Cells

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ABSTRACT: A conceptually new polymer electrolyte for dye-sensitized solar cells is reported and investigated. The benefits of using this type of electrolyte based on ionic liquid mixtures (ILMs) and room temperature ionic liquids are highlighted. Impedance spectroscopy and transient electron measurements have been used to elucidate the background of the photovoltaic performance. Even though larger recombination losses were noted, the high ion mobility and conductivity induced in the ILMs by the added polymer result in enhanced overall conversion efficiencies.

INTRODUCTION

Due to the constantly increasing global energy demand, much attention has been devoted to the conversion of the abundant solar energy to electricity. In this context, another vital motive to pursue research on renewable energy sources is the ambition to reduce the environmental hazards associated with today’s predominant energy technologies. Direct conversion of solar energy to electricity, for example, by photovoltaic devices, constitutes an attractive alternative for the future. Dye-sensitized solar cells (DSSCs) were converted into a promising and alternative green technology for renewable energy in 1991 through a seminal paper by Grätzel and O’Regan, with the main advantages being potential cost-effective fabrication and good conversion efficiencies.1,2 In recent reports, photoconversion efficiencies over 12% have been obtained by using organic solvent-based electrolytes together with a liquid electrolyte based on the ubiquitous iodide/triiodide redox couple.3–5 The use of biosourced aqueous gel electrolytes and newly designed bifacial quasi-solid state electrolyte-based DSSCs were also reported.5,7 Even higher conversion efficiencies have been obtained by use of metal-complex redox systems approaching 15%.5,8

Room temperature ionic liquids (ILs) present some very promising features that make them highly attractive for DSSC applications. ILs have been widely promoted as “green solvents” mainly because of their typically extremely low vapor pressures and have recently attracted considerable attention also because of their excellent chemical and electrochemical stability, nonflammability, and high ionic conductivity.9–17 A recent report shows that mixtures between molten salts and molecular solvents exhibit unique physical and chemical properties differing from their component properties. They also contribute new properties, such as low molecular vapor pressure, wide electrochemical window, and low viscosity. This type of fluid is denoted “incompletely solvated ionic liquid mixtures (ILMs),” or simply “ionic liquid mixtures” (ILMs), and are characterized by the molecular component being present in such low ratios that there is not sufficient number of molecules to completely solvate the ions of the salt. Of course, any such mixture may have complex structures on the nanometer scale, but nonetheless they contain too few molecules to physically separate the ions from each other.16,19

As reported elsewhere, the strategy to achieve an ionically highly conductive electrolyte is to dissolve a suitable polymer in electrolyte salts yielding high conductivity as compared with their parent components. In this work, we have chosen poly(vinylidine fluoride) (PVDF) as the polymer dopant due to its favorable chemical properties, such as chemical stability, high dielectric constant, and resistance to UV radiation. The above polymer in electrolyte salts, also known as polymer-in-salt electrolytes (PISSs), typically increases the ionic mobility and the number of carrier ions.20 This effect can be traced to an increase in ion-pair dissociation, although the exact mechanism is currently unclear.
In the present work, we aim to harvest the beneficial effects of the ILs, ILMs, and PISs to prepare a conceptually new class of electrolyte for DSSCs. The ion-conducting salt plays an important role for the ionic conduction and a polymer matrix works as a rigid framework, also promoting ion dissolution or ion polarization. The resulting new type of electrolyte shows many desirable properties, such as low vapor pressure, chemical stability, low viscosity, and high number of ionic charge carriers. In this work, the main focus has been to investigate and compare this new type of electrolyte to those based on ILs or ILMs in DSSC devices.

RESULTS AND DISCUSSION

Photovoltaic Performance. Figure 1 shows the $I–V$ curves of the DSSCs containing the electrolytes IL, IM, and IP, displaying efficiencies of 3.6, 6.6, and 6.9% at 1 sun light intensity, respectively. The $I–V$ characteristics of the respective cells are given in Table 1. The cells containing the electrolytes IM and IP show considerably higher current than those containing the IL electrolyte. The slope of the $I–V$ curve from the photovoltage axis can be used to estimate the cell series resistance (and thus conductance), and from this the total number of available ions for charge transport in the cells can be estimated; hence, more ions are available for charge transport in the IM- and IP-based cells, and consequently these cells show higher photocurrents. The fabricated cells were stable without any notable degradation even after 1000 h storage (dark conditions), which is an indication that they are chemically stable.

The incident photon-to-current conversion efficiencies (IPCEs) for the IL-, IM-, and IP-based cells are shown in Figure 2. The IPCE spectrum for IP-based cells shows a maximum value of 81% just below 450 nm. The IL- and IM-based cells display a maximum value from 375 to 550 nm. The light absorption by triiodide at shorter wavelengths can be noted as a dip in the IPCE spectrum.

Electrochemical Impedance Spectroscopy (EIS) Studies. The Nyquist plots of the DSSCs based on the electrolytes IP, IM, and IL are shown in Figure 3 and selected characteristics are assembled in Table 2. The complex impedance is defined as $Z^*(\omega) = Z'(\omega) - jZ''(\omega)$, where

| Table 1. Current–Voltage Characteristics for Different DSSC Cells (Six Cells Were Made of Each Type) |
|---------|---------|---------|---------|---------|
| types   | $V_{oc}$ (mV) | $J_{sc}$ (mA cm$^{-2}$) | FF | $\eta$ (%) |
| IL      | 815      | 6.96    | 0.64   | 3.6   |
| IM      | 830      | 11.1    | 0.71   | 6.6   |
| IP      | 810      | 11.9    | 0.71   | 6.9   |

| Table 2. Parameters Obtained from the Impedance Spectroscopic Studies |
|----------------|----------------|----------------|----------------|
| cells | resistance ($R_\infty$, $R_{rec}$, $R_d$/Ω) | electron lifetime (ms) | conductivity ($10^{-5}$ S cm$^{-1}$) | viscosity (cP) |
| IL    | 6       | 8.0              | 5.43            | 24   |
|       | 37      |                   |                 | 68   |
| IM    | 10      | 8.8              | 6.36            | 21   |
|       | 32      |                   |                 | 13   |
| IP    | 8       | 7.6              | 6.00            | 22   |
|       | 33      |                   |                 | <1   |
Z(ω) = |Z|cos θ and Z*(ω) = |Z|sin θ are the real and the imaginary parts of the complex impedance, respectively, and ω = 2πf is the angular frequency. The impedance spectra were recorded for the DSSCs under open-circuit condition with an applied bias voltage. The curves were fitted by an electrochemical circuit model with the help of the ZView software.21

The three types of DSSCs displayed the resistance of the fluorine-doped tin oxide (FTO) films (R_e) and external effects as a serial resistance. Going from the left to the right (from high frequency to low frequency) in Figure 3, the typical interpretation of the features involves a first semicircle that represents the interface resistance (R_int) corresponding to the charge transfer between the counter electrode and the electrolyte in the high-frequency region from 100 kHz to 550 Hz. As seen in the zoomed plot, the resistance (R_int) for the IL-based cells is comparatively small, whereas it is considerably larger for the IM-based cells, almost by a factor of 2. The presence of 3-methoxypropionitrile in the electrolyte IM and PVDF in the electrolyte IP influences the electrolyte–electrode interface, as inferred by the higher R_int for the IM-based, slightly lower value for the IP-based, and very low for IL-based cells. Interestingly, the IP-based DSSCs show a lower R_int than the cells based on IM. Similar observations were reported in our previous work,22 where the R_int for DSSCs containing the solvent-containing electrolyte (CM1) is comparably higher than those containing the added polymer (CP1). It is clear that dilution of the ionic liquid retards the charge-transfer process at the counter-electrode.

The second semicircle in the middle frequency region, from about 550 to 4 Hz, offers information about the electron recombination loss resistance (R_rec) corresponding to the charge transfer at the working electrode and electrolyte interface, and the chemical capacitance produced by the accumulation of electrons in the TiO2 film and cations at the TiO2–dye interface (Cμ).23 The R_rec of the DSSCs decreases in the order IL ≫ IM ≈ IP, but they are all very similar and there should be no significant difference in electron recombination loss for the three types of electrolytes in the DSSCs.

The resistance in the low-frequency region (R_d), from 4 Hz to 10 mHz, relates to the diffusion impedance of the electrolyte. The diffusion resistance decreases in the order IL ≫ IM ≈ IP. This clearly indicates that the ionic mobility is lower in the IL electrolyte than in the other two. Another way of presenting the complex impedance is by displaying the frequency response of the modulus of impedance and phase angle, as shown in the Supporting Information (Figure S1), for the DSSCs based on the IL, IM, and IP electrolytes. The conductivity (σ) and electron lifetime (τ) were calculated according to the procedures described in an earlier report22 and the values were tabulated (Table 2). The conductivity of the electrolyte solution is proportional to the number of charge carriers and also inversely proportional to the medium viscosity,24 relations that match well with our viscosity studies (Table 2). The difference observed between IP- and IM-based cells is difficult to distinguish from the above results and further analysis based on alternating current (AC) conductivity was used to identify the different conduction mechanisms in the DSSCs.

**Transient Measurements.** The electron lifetime (τ_e) is shown as a function of voltage in Figure 4. The electron lifetime was determined at open-circuit conditions using a modulated light source. The changes in electron concentration are mainly affected by recombination because the current and bias light are fixed. The time constant found by the voltage decay can be used to calculate the electron lifetime.25 At zero current, the IM- and IL-based cells show similar behavior, whereas the IP-based cells show shorter electron lifetimes, indicating faster electron loss reactions. These results are consistent with the EIS data, where the IP-based cells display larger recombination losses. However, because of the higher electrolyte conductivity, the cells anyhow are the ones performing the best in overall conversion efficiency.

The extracted charge Q_rec as a function of Voc for the DSSCs based on the IL, IM, and IP electrolytes are shown in Figure 5.

It is clearly noted that cells based on the IM and IP electrolytes exhibit very similar extracted charges at higher voltages, whereas the IL-based TiO2 films are slightly less charged. At lower voltages (essentially corresponding to lower light intensities), the IP-based cells clearly outperform the others, which is in accordance with the electron lifetime results. These results also match the higher J_sc obtained in the I–V measurements.
The electron lifetime ($\tau_e$) is shown as a function of total charge in Figure 6. The IP- and IM-based DSSCs show longer electron lifetimes than IL-based ones. This is in good agreement with the EIS studies, which indicate slightly larger recombination losses for the IP- and IM-based cells. Transport times are nearly the same for all types of cells at lower current, which indicates that the three electrolytes have the same effect on TiO$_2$ with respect to the electron conduction processes. The transport time with respect to short-circuit current for the IP-, IM-, and IL-based cells are shown in Figure S5.

The overall beneficial factor of the addition of polymer to this type of electrolyte is that it very clearly yields an increase in photocurrent. This increase is likely to be caused by an increase in the ion mobility in the bulk electrolyte.

### CONCLUSIONS

In this present work, we investigated the merits of using a conceptually new electrolyte. The IM-based cells rendered a conversion efficiency of 6.6% and those of IP-based cells 6.9%. The use of both ILMs and polymer addition gives a boost in efficiencies. The current results encourage further studies on the effects of polymer addition for enhancing the DSSC efficiency.

### EXPERIMENTAL METHODS

**Materials and Methods.** All of the chemicals were used without further purification. Poly(vinylidene fluoride) (PVDF) (Alfa Aesar), ionic liquid (ethyl-methylimidazolium dicyanam ide (EMIDCA)) was obtained from Merck, methyl benzimidazole and 3-methoxypropionitrile from Sigma-Aldrich, and guanidine thiocyanate (GuSCN) and 1-propyl-3-methyl-imidazolium iodide (PMII) from Alfa Aesar. The dye D35 was obtained from Dyenamo AB, Sweden. The TiO$_2$ pastes (DSL 18NR-T and WER2-O) were obtained from Dyenamo Ltd.

**Electrolyte Preparation.** Poly(vinylidene fluoride) was dissolved in 3-methoxypropionitrile (1.5 wt %) and stirred at 100 °C for 2.5 h. After cooling to room temperature, the following recipes were employed for the preparation of the electrolytes for comparative studies, where the polymer solution is used in the third recipe:

**Recipe 1:** ionic liquid (EMIDCA) 200 µL, I$_2$ 0.2 M, GuSCN 0.1 M, PMII 1 M, and 0.5 M methyl benzimidazole (IL).

**Recipe 2:** ionic liquid (EMIDCA) 100 µL, I$_2$ 0.2 M, GuSCN 0.1 M, PMII 1 M, and 0.5 M methyl benzimidazole in 100 µL 3-methoxypropionitrile (IM).

**Recipe 3:** ionic liquid (EMIDCA) 100 µL, I$_2$ 0.2 M, GuSCN 0.1 M, PMII 1 M, and 0.5 M methyl benzimidazole in 100 µL in PVDF—MPN solution (IP).

The viscosities of the electrolytes were determined using a Brookfield (CAP 2000+) viscometer.

**Electrode Preparation.** The glass substrates with a transparent conductive film (fluorine-doped tin oxide (FTO), TEC15; Pilkington) were cleaned in an ultrasonic bath following the consecutive steps: soap water, acetone, and ethanol; 20 min in each step. Then, the FTO substrates were pretreated by immersion into a 40 mM aqueous TiCl$_4$ solution at 70 °C for 30 min and washed using water and ethanol. The TiO$_2$ screen-printing employed a DSL 18NR-T (Dyesol Ltd.) paste, and after the screen-printing procedure, the glass electrodes were stored in an atmosphere of EtOH for 5 min and then dried at 120 °C for 5–6 min. Finally, the procedure was repeated for a second time to obtain a uniform film. Areas of 0.25 cm$^2$ of photoactive TiO$_2$ films were screen-printed. After cooling of the electrodes, a light-scattering TiO$_2$ layer (WER2-O; Dyesol Ltd.) was printed. Thereafter, the electrodes were gradually fired in the ambient atmosphere in three steps of temperature increase; 180 (10 min), 325 (10 min), 420 (10 min), and 450 °C (30 min). After sintering, the electrodes were again treated with a 40 mM TiCl$_4$ solution in water at 70 °C for 30 min and washed according to the previously described process. The substrates were again sintered at 500 °C (30 min; thickness 13 µm, Veeco Dektak 3). After the final step of sintering, the electrodes were allowed to cool to 80 °C and directly immersed into a bath of dye at 0.25 M D35 in ethanol and left over night. Platinized counter electrode substrates (TEC15; Pilkington) were prepared by deposition of 20 µL of 4.8 mM H$_2$PtCl$_6$ 2-propanol solution to a predrilled FTO glass substrate; the platinized electrodes were sintered in ambient atmosphere at 400 °C for 30 min.

**Solar Cell Assembly and Characterization.** The working electrodes and the counter electrodes were combined into a sandwich-type device by a 25 µm thick Surlyn thermoplastic frame (Solaronix), and the electrolyte was immersed via a predrilled hole through the glass plate of the counter electrode. Finally, the hole was sealed using Surlyn and a glass coverslip.

A Keithley 2400 source meter was used to record the current–voltage (I–V) characteristics in combination with a Newport solar simulator (model 91160) based on AM 1.5 G spectral distribution. Calibration was based on a certified reference solar cell (Fraunhofer ISE) at an intensity of 1000 W m$^{-2}$, alternatively using a neutral density filter at 100 W m$^{-2}$. Scattered light from the edge of the glass electrodes of the dyed TiO$_2$ layer was reduced using a mask of the size 7 × 7 mm$^2$. This procedure also reduces the contribution from light outside the active area. Incident photon-to-current conversion efficiencies (IPCEs) were monitored by monochromatized light from a system comprising a xenon lamp, monochromator, suitable filters, and a potentiostat. A certified reference solar cell (Fraunhofer ISE) was used for calibration. Electron lifetimes for fully operational solar cells were determined using a green light-
emitting diode (1 W; Luxeon) as the light source. A 16 bit data acquisition board (DAQ National Instruments) combined with a current amplifier (Stanford Research SR570) were used to record the voltage/current traces. Charge extraction was determined following a procedure in which the solar cell was illuminated during 5 s under open circuit, after which the light was switched off and the voltage decay was monitored at a specific voltage V. At this voltage V, the cell was short-circuited, and the current was recorded during 10 s. Integration provided the charge, Q_{ac} (V). Electron lifetimes were monitored following the photovoltage response after a small perturbation of the light intensity.

**Electrochemical Impedance Spectroscopy (EIS).** An Autolab PGstat12 potentiostat was used to record the electrochemical impedance spectra (EISs). The potentiostat was equipped with an impedance module allowing a frequency range of 10^{-2} to 10^{7} Hz, at 20 mV AC bias under light illumination in combination with an applied bias potential. The ZView program was used to model the experimental data.

### ASSOCIATED CONTENT

#### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.7b00925.

Phase angle and modulus impedance for IL, IP, and IM cells (Figure S1); normalized conductivity plot for IL, IP, and IM cells (Figure S2); exponent n as a function frequency for IL, IP, and IM cells (Figure S3); relaxation time for IL, IP, and IM cells (Figure S4); transport time with respect to short-circuit current for the IP-, IM-, and IL-based cells (Figure S5) (PDF)

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**Notes**

The authors declare no competing financial interest.

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