

Research Article

High-Efficiency Glass and Printable Flexible Dye-Sensitized Solar Cells with Water-Based Electrolytes

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The performance of a flexible and glass dye-sensitized solar cell (DSSC) with water-based electrolyte solutions is described. High concentrations of alkylamidazoliums were used to overcome the deleterious effect of water and, based on this variable, pure water-based electrolyte DSSCs were tested displaying the highest recorded efficiency so far of 3.45% and 6% for flexible and glass cells, respectively, under a simulated air mass 1.5 solar spectrum illumination at 100 mWcm^{-2} . An improvement in the J_{sc} with high water content and the positive impact of GuSCN on the enhancement of the performance of pure water-based electrolytes were also observed.

1. Introduction

Dye-sensitized solar cells (DSSCs) are considered so far to be the best low cost alternative to silicon photovoltaic cells [1–3] demonstrating an efficiency of up to 12% [4] and have been intensively studied since their discovery in 1991 [5]. The underlying process for this technology relies upon a sensitizing dye adsorbed onto the surface of a very thin, porous, ceramic-like sponge made of titanium dioxide (TiO_2), a commonly available material. The dye traps light and then uses this energy to inject an electron into the TiO_2 . These electrons become available for producing useful electricity when the TiO_2 is connected through an external load, such as a battery, light, or other electrical devices, thus closing the circuit so that the electrons are returned back to the dye. The flexible DSSC modules are made of thin and flexible polymers or foils, which are tough, durable, safe to use, and environmentally friendly since the electricity produced is clean and the manufacturing processes are based on abundant, recyclable materials. However, water can permeate into the device relatively rapidly due to the flexible plastic design as opposed to glass. Permeation can be reduced by barrier layers, but this increases the cost. Water was frequently used as an electrolyte in early DSSCs but was abandoned in favour

of organic solvents that gave much higher cell efficiencies. In the past, many reports were of the opinion that water was poisonous for DSSCs and that the presence of even a slight trace of water in the electrolyte would be detrimental to the cell performance and would reduce cell stability either by dye detachment [6], by formation of iodate [7], or by a decrease in electron life-time [8]. Recently, however, it has been thought that water-based DSSCs could be relevant to DSSC development since cells, properly optimized to work with water, can give reasonable efficiencies [9] and display other advantages. The highest published efficiencies for 100% water-based electrolyte cells made using 1 sun have increased slightly from 1.1% [10] to 2.4% [9] and to 4% [11], and recent work based on water gel polymer shows that efficiencies of 2.1% [12] and 4.78% [13] were achieved. These results could justify the return to reexamining water as a less expensive and more environmentally friendly alternative for DSSCs. To our knowledge, while the focus was previously on glass cells, 100% water-based electrolytes remained untested at 1 sun for printable flexible DSSCs. Law et al. [9] reported that a water-based organic electrolyte with the following formulation: 2 M 1-propyl-3-methylimidazolium iodide (PMIM I), 0.05 M iodine, 0.1 M guanidinium thiocyanate (GuSCN), and 0.5 M tert-butylpyridine (TBP), functioned without efficiency loss

with up to 40% water fraction but it decreased significantly at 60% water fraction. Another study [11] showed that for a pure water electrolyte with a high concentration of guanidinium iodide GuI 8 M (its stability has not yet been demonstrated and it is not commercially available, thus increasing the cost) it was possible to achieve an efficiency of 4% by adding more additives to the electrolyte formulations to improve the wetting of the TiO₂. Out of interest, we prepared pure water electrolytes with a concentration of different alkylimidazolium derivatives slightly higher than 2 M varying from 3 M to 4 M (1.4 M for BMIM-I for our standard electrolyte) in order to overcome the deleterious effect and by doing so we were able to avoid the phase segregation and to achieve an efficiency of 3.45% and 6% for flexible and glass cells, respectively, in 100% water-based DSSC. This work is presented in two parts. In the first part, we test water content electrolytes for flexible cells only since glass cells were already tested in a previous study. We present detailed measurements of cell characteristics versus water content to observe the influence of water on flexible cells. In the second part, we test both glass and flexible cells with pure water electrolytes with different formulations; DSSCs show an improvement in performance compared to those with aqueous and organic electrolytes. Also presented in this work is a study of the life-time performances for water-based DSSCs under soaking illumination.

2. Experimental

1-Propyl-3-methylimidazolium iodide PMIM-I, 1-butyl-3-methylimidazolium iodide BMIM-I, 1-propyl-3-methylimidazolium bis-(trifluoromethanesulfonyl)-imide PMIM-TFSI, 1-butyl-3-methylimidazolium bis-(trifluoromethanesulfonyl)-imide BMIM-TFSI, 1-ethyl-3-methylimidazolium bis-(trifluoromethanesulfonyl)-imide EMIM-TFSI, and N-butylbenzimidazole BBI were purchased from BASF; 4-tert-butylpyridine TBP, guanidinium thiocyanate GuSCN, iodine, sulfolane, and other solvents were purchased from Sigma-Aldrich; deionized water was purchased from ReAgent (conductivity < 1 μ S/cm); the ruthenium dye Z907, titanium foil, scattering TiO₂ nanoparticle paste, plastic counter electrode platinum-sputtered ITO/PET (PET: polyethylene terephthalate)/15 Ω /cm², glass platinum-sputtered ITO/FTO (FTO: fluorine-doped tin oxide)/15 Ω /cm², 4-guanidino-butyric acid (GBA), hydrogen hexachloroplatinate (IV) hydrate (H₂PtCl₆·H₂O), iodine, and 2-propanol were offered by G24i.

Glass cells were fabricated using standard procedures. The TiO₂ films were made from colloidal solutions using a screen printing method and then heated to 500°C for 30 minutes. Two layers were deposited successively: a 4 μ m thick transparent layer followed by a 4 μ m light-scattering layer of nanocrystalline TiO₂. Dyeing was done using 24 mM:24 mM :Z907:GBA solutions in an alcohol-based solvent for 5 minutes. The counter electrodes were fabricated by applying 8 mM H₂PtCl₆ in 2-propanol to the FTO glass, followed by heating in an oven at 500°C for 30 minutes. Cell assemblies were formed by sealing the counter

electrodes to the TiO₂ electrode with Surlyn (Dupont) at 100°C for 1 minute. The corresponding electrolyte was introduced through two small holes, previously drilled through the counterelectrode, which were then sealed with Surlyn. Photocurrent versus voltage characteristics were measured with a Keithley 2602 sourcemeter and a LED solar simulator illuminated. The irradiated area of the cell was 0.3 cm².

Flexible cells were fabricated using standard procedures. The TiO₂ nanoparticles and titanium foil were offered by G24i. Dyeing was done using 24 mM:24 mM :Z907:GBA solutions in an alcohol-based solvent for 15 minutes. A platinum-sputtered ITO/PET was used as counter electrodes. Cell assemblies were formed by sealing the counter electrodes to the TiO₂ electrode with Bynel at 150°C for 10 seconds. The corresponding electrolyte was introduced through the edge of the cell by capillarity using a needle. Photocurrent versus voltage characteristics were measured with a Keithley 2602 sourcemeter and a LED solar simulator illuminated. The irradiated area of the cell was 8.1 cm².

Symmetrical cells or dummy cells were prepared by the following methods: two rectangles from the counter electrode CE film approximately 25 mm \times 35 mm were cut and stacked together by a thick square surlyn 25 mm \times 25 mm in size and a 12 mm diameter hole was punched using syringes. Finally, the water-based electrolytes were added into the holes using desecrator and vacuum pump and the holes were covered by silicone paste.

Cell I-V characteristics were measured using illumination under AM 1.5 condition (100 mW/cm²) from LED (Phillips LUXEON Rebel LXML-PWC2, Cool white). These LEDs cover the absorption spectra and mimic sun spectra for G24i technology. The IV data was collected from the Keithley 2602.

Life-time studies were carried out at long-term illumination at 1 sun with the Halogen lighting (Sylvania Dichroic 5130 12 V/50 W/60°C).

3. Results and Discussion

The aim was to completely remove the sulfolane solvent from the iodine/tri-iodide electrolyte and to replace it by gradually adding water. Samples were produced with different water percentages 0, 5, 10, 20, 40, 60, 80, and 100% and the following ionic liquid additives BMIM-I 1.4 M, PMIM-TFSI 0.7 M, BBI 0.4 M, and I₂ 0.1 M were used. For comparison between the performances of different water-based electrolytes, the concentrations of the additives remained constant. Iodine was added to BMIM-I first to ensure that all the iodine ions were dissolved completely into the electrolyte; PMIM-TFSI, BBI, water, and sulfolane were added to the solution and mixed at room temperature for 1 hour and then heated at 40°C for 1 hour. It was noted that sulfolane solidifies at room temperature; for this reason it was heated at 40°C to be used as a liquid for all the samples. After completing the formulation, red-brown solution electrolytes were obtained. It was observed that at 40–100% of water the solutions were not homogeneous (see Supporting Information, S1, available online at <http://dx.doi.org/10.1155/2014/426785>) due to nonmiscibility between water and the additives.

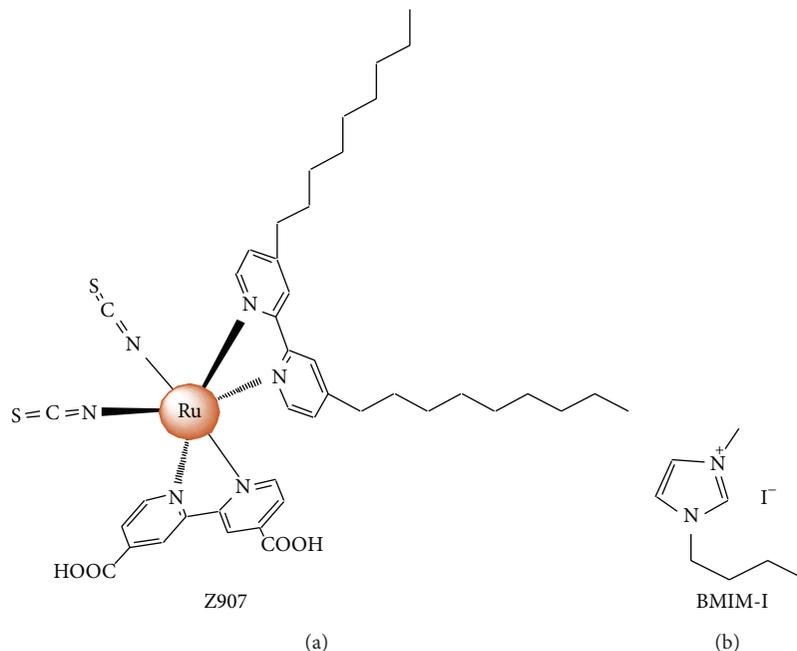


FIGURE 1: Chemical structures of Z907: *cis-bis(isothiocyanato)(2,2'-bipyridyl-4,4'-dicarboxylato)(4,4'-di-nonyl-2'-bipyridyl)ruthenium(II)*; and an example of alkylimidazolium BMIM-I: *1-butyl-3-methylimidazolium Iodide*.

The solar energy-to-electricity conversion efficiency, η (%), of solar cells was estimated from the following equation:

$$\eta = \frac{(J_{sc} \times V_{oc} \times FF)}{I_0} \times 100, \quad (1)$$

where I_0 is the photon flux (ca. 100 mW/cm^2 for AM 1.5 G), J_{sc} is the short-circuit current density under irradiation, V_{oc} is the open-circuit voltage, and FF is the fill factor. The hydrophobic dye Z907 (Figure 1) was selected in this study to avoid its desorption into the water electrolyte. Alkylimidazoliums were selected as an iodide source for these studies due to their ability to enhance the conductivity of an electrolyte containing the redox couple of I^-/I_3^- and to increase the electron diffusion coefficient in TiO_2 [14, 15].

The characterization of all samples was carried out with the help of FTIR Spectroscopy *Smart Germanium* integrated with *OMNIC* software. The IR spectra of all the samples (Supporting Information, S1) present a water band at 3500 cm^{-1} with the absorbance increased by increasing the water quantity in the electrolyte samples. The bands' regions $500\text{--}1500 \text{ cm}^{-1}$ and 3000 cm^{-1} correspond to the additives present in the electrolytes. No changes were observed in the IR except that the absorbance of the electrolyte bands decreases by increasing water due to the problem of nonmiscibility between water and the additives present in the electrolyte. Water does not show a chemical reaction with the electrolyte additives especially BMIM-I, PMIM-TFSI, and BBI.

A series of cells with sulfolane and different water contents were fabricated; the photovoltaic characteristic of the water content electrolyte is summarized in Table 1 and Figure 2. Increases in photovoltaic performances by adding a small amount of water were demonstrated by Mikoshiba

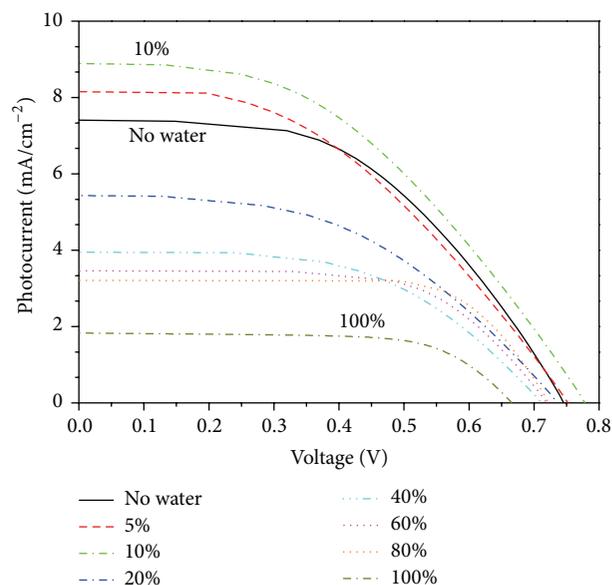


FIGURE 2: I - V curves obtained from a water-based flexible cell with 1 sun illumination measured in the BMIM-I 1.4 M, PMIM-TFSI 0.7 M, BBI 0.4 M, I_2 0.1 M, water %, and sulfolane %.

et al. [16]; our results show that the 10% water cell exhibits a higher efficiency than the other water-added cells. The enhancement of V_{oc} afforded by the addition of 10% water is due to the reduction of the back electron transfer from the TiO_2 conduction band to the I_3^- ions in the electrolyte [8], but it was found that the short circuit current J_{sc} and, therefore, the efficiency were limited by diffusion in cells with

TABLE 1: Photoelectrochemical data obtained from a water-based flexible cell with 1 sun illumination measured in the BMIM-1 1.4 M, PMIM-TFSI 0.7 M, BBI 0.4 M, I_2 0.1 M, water, and sulfolane.

Water content [%]	Sulfolane content [%]	J_{sc} [mA/cm ²]	V_{oc} [V]	FF	Efficiency [%]
0	100	7.41	0.75	0.46	2.56
5	95	8.15	0.75	0.44	2.69
10	90	8.89	0.77	0.44	3.01
20	80	5.43	0.73	0.48	1.90
40	60	3.95	0.71	0.54	1.51
60	40	3.46	0.72	0.63	1.57
80	20	3.21	0.72	0.71	1.64
100	0	1.83	0.67	0.70	0.86

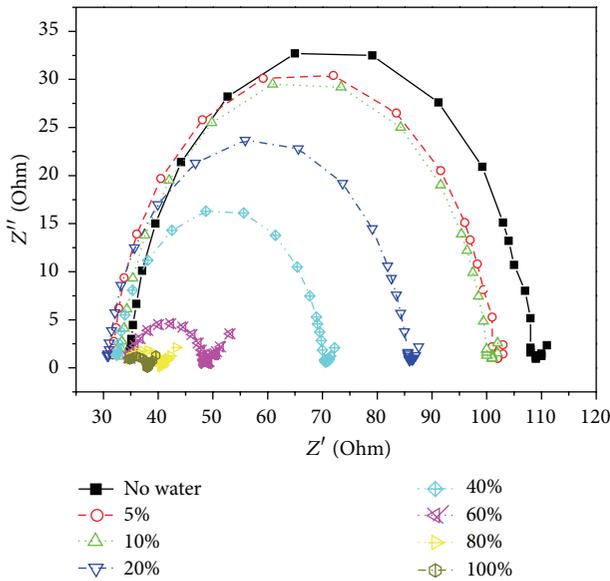


FIGURE 3: Nyquist plots of electrochemical impedance spectra containing different water-based electrolytes for symmetrical cells in dark conditions. The plots were measured with two platinum-sputtered ITO/PET counter electrodes for each electrolyte and recorded over the frequency range of $0.01\text{--}10^4$ Hz at $V_{oc} = 0$ V with an ac amplitude of 10 mV.

a high water content. Note that the fill factor increases slightly with water content. It is known that the fill factor depends on the resistance of the TiO_2 electrode, electrolyte, and counter electrode, as observed by Jung et al. [8]. The increase in the fill factor in the case of the cell with high water content is attributed to its charge transfer resistance.

To understand the decrease in J_{sc} , the electrochemical impedance spectroscopy was carried out. Figure 3 shows the Nyquist plots of the electrochemical cells consisting of two Pt-coated ITO/PET counter electrodes with different water contents in the electrolyte. The corresponding charge transfer resistances, R_{ct} , obtained by fitting the plots with *IviumSoft* software based on a standard Randles equivalent circuit for the frequencies from 100000 Hz to 0.01 Hz, are listed in Supporting Information S2. The values of the R_{ct} refer to the resistance against transfer from the counter electrode

to I_3^- . The results imply that the R_{ct} of the water-added DSSC is decreased compared to that of the nonwater DSSC (Figure 3). A lower charge transfer resistance implies a more facile electron transfer from counter electrode to I_3^- ions [8], but, contrary to that, the J_{sc} values of water-added DSSCs decreased. Furthermore, the viscosity of each water-added electrolyte was measured with the help of *Bohlin Gemini* integrated with the *Bohlin* software. The table (Supporting Information, S2) reveals that the addition of water decreases the viscosity of the water-added electrolyte. This decrease is considered to be favorable for the movement of the ions. The observation of this phenomenon with the addition of water is in agreement with the previous studies [8]. The decrease in J_{sc} and the efficiency of the cells with high water content may be explained by several factors; for instance, the equilibrium constant at 25°C for the tri-iodide formation is much weaker in water than in an organic solvent [17]. Another study suggests that the injection of electrons from the dye into the TiO_2 depends on the energy level matching between the excited dye molecules and the conduction band edge. It was found that the frontier electronic structure was important for the charge transfer in DSSC and that the reduction of the dye in a working DSSC involved an electron transfer from the redox couple to the oxidized dye [18]. According to Murakami et al. [10], the surface of the TiO_2 showed that hydrophilicity was not high enough to allow efficient permeation of water redox electrolyte into the mesoporous structure due to the high surface tension of water compared with an organic electrolyte, whilst Law et al. [9] demonstrated that the loss of photocurrent at 1 sun light intensity is mainly due to a reduced current carrying capability of the electrolyte in the pores, not to fundamental problems with electron transfer kinetics at the TiO_2 /dye/electrolyte interface. The pH studies were carried out (Supporting Information, S2) which clearly showed that it slightly decreases with increasing water quantities; thus, the acidity of the solutions increases. The TiO_2 band edge has been shown to shift according to the Nernstian potential dependence of 60 mV per pH unit in solution [19]. Other studies [16, 20] show that the conduction band edge of nanoporous TiO_2 films shifts positively when water is added to an aprotic solution as a result of the decrease in the number of dye molecules limiting the flux of the injected electrons from the excited states of the dye and causing the decrease of J_{sc} .

TABLE 2: Photoelectrochemical data obtained with the 100% water-based electrolyte for flexible and glass cells at 1 sun illumination.

Electrolyte formulation	Water content [%]	Flexible cells				
		Electrolyte composition	J_{sc} [mA/cm ²]	V_{oc} [V]	FF	Efficiency [%]
Standard	0	1.4 M BMIM-I, 0.7 M PMIM-TFSI, 0.4 M BBI, 0.1 M I ₂	7.41	0.75	0.46	2.56
IC100	100	4 M PMIM-I, 0.2 M GuSCN, 0.1 M I ₂ , 1.2 M tBP	7.45	0.77	0.60	3.45
PE100	100	3.8 M PMIM-I, 0.7 M EMIM-TFSI, 0.1 M I ₂ , 0.4 M BBI	6.95	0.74	0.56	2.88
PB100	100	3.6 M PMIM-I, 0.7 M BMIM-TFSI, 0.1 M I ₂ , 0.4 M BBI	7.01	0.74	0.59	3.05
Glass cells						
Standard	0	1.4 M BMIM-I, 0.7 M PMIM-TFSI, 0.4 M BBI, 0.1 M I ₂	9.97	0.78	0.53	4.12
IC100	100	4 M PMIM-I, 0.2 M GuSCN, 0.1 M I ₂ , 1.2 M tBP	11.01	0.79	0.69	6.00
PE100	100	3.8 M PMIM-I, 0.7 M EMIM-TFSI, 0.1 M I ₂ , 0.4 M BBI	9.48	0.75	0.65	4.62
PB-100	100	3.6 M PMIM-I, 0.7 M BMIM-TFSI, 0.1 M I ₂ , 0.4 M BBI	9.97	0.76	0.67	5.07

Long-term stability tests were carried out on the flexible cells with various water-based electrolytes during 1,000 hours under continuous illumination at 1 sun. Life-time results are shown in Supporting Information, S3. It was observed that the J_{sc} for all water-based electrolytes was stable with a slight increase in the first 200 hours and stabilized thereafter until 1,000 hours. Upon completion of the life-time studies, the water-based flexible cells were examined for dye detachment using UV-Vis spectroscopy (Supporting Information, S4). No dye desorption was detected in the cells after 1000 hours under light soaking.

In the first instance, studies were carried out on electrolytes containing different water percentages with varying quantities of sulfolane from 100 to 0%. The pure water-based electrolyte did not prove to display a higher efficiency, notably 0.86% (Table 1). Following on from this, we were able to enhance this result by simply increasing the amount of alkylimidazolium to reduce the deleterious effect of water. For this reason, sulfolane, as an organic solvent, was eliminated from the electrolyte and only water was used. Different pure water-based electrolytes were prepared (Table 2): IC100, PE100, PB100, by using PMIM-I, as alkylimidazolium derivatives. The alkylimidazolium-bis-(trifluoromethanesulfonyl)-imide (BMIM-TFSI, EMIM-TFSI) and BBI were tested as derivatives for different electrolytes and GuSCN and TBP were selected for IC100 formulation. The added quantity of the iodine [I₂] = 0.1M remained constant for all the formulations. It should be noted that the concentration of the additives for the reference electrolyte was as follows: BMIM-I 1.4 M, PMIM-TFSI 0.7 M, I₂ 0.1 M, BBI 0.4 M in sulfolane.

Water-based electrolyte for all formulations gives the best performance for flexible cells, with the photocurrent being about few mA/cm² more or less higher than the standard electrolyte; however, the efficiencies are within the same range with a maximum of 3.45% and 6% for flexible and glass cells, respectively. Fill factor is higher for all samples with pure water. Comparatively, pure water electrolytes tested in glass cells (Table 2) showed a high performance because the illumination at 1 sun was carried out at the back of the cell and directly on the TiO₂ electrode which is not possible with the design of the flexible cells; furthermore

the conductivity in the counterelectrode of the glass cells is higher than the flexible cells. The efficiencies for water-based electrolytes in glass cells displayed the highest recorded efficiency, to date, of 6% whilst a current of 11 mA/cm² was recorded with a high voltage of 790 mV. It has been shown that pure water does not wet Z907 dye TiO₂ [21]; however, our study was carried out without the need for any surfactants like chenodeoxycholic acid as described by [22] or Triton X-100, as a nonionic surfactant was used to enhance the water solubility [8]. However, we were able to notice the role of GuSCN to improve J_{sc} and the efficiency for the IC100 formulation. Guanidinium is said to be a chaotropic ion, essentially reducing the strength of hydrophobic interactions, therefore possibly increasing wetting and diffusion limited current [23]. At the same time, the thiocyanate ion also binds iodine strongly and may thus reduce free iodine and increase collection efficiency. A previous study has found that GuSCN could improve the solar performance by slowing the surface recombination [24]. Gratzel reported that the addition of the guanidinium cation to the electrolyte could control the self-assembly of the dye at the TiO₂ interface and suppress the dark current [3]. Zhang et al. demonstrated the positive effect of GuSCN on DSSC to improve the performance and stability [25]. We did notice similar performances in water with GuSCN for the IC100 formulations in flexible and glass cells.

Figure 4 presents the I - V comparison performance between a standard electrolyte and a pure water IC100. High efficiencies of 3.45% and 6% were recorded for flexible and glass cells, respectively, with better performance for glass cells as previously mentioned. A significant improvement was observed in FF for the pure water electrolyte; J_{sc} also increased by adding GuSCN. There did not appear to be change in the V_{oc} although some studies showed that V_{oc} decreases when the pH is acidic, which could be explained by the use of a high concentration of alkylamidazolium which neutralizes the acidity effect, thus possibly resulting in a slight improvement in the V_{oc} by a few millivolts.

To complete this study, we subjected a selection of IC100 for glass water-based DSSCs to light soaking condition. Figure 5 shows long-term stability under continuous AM

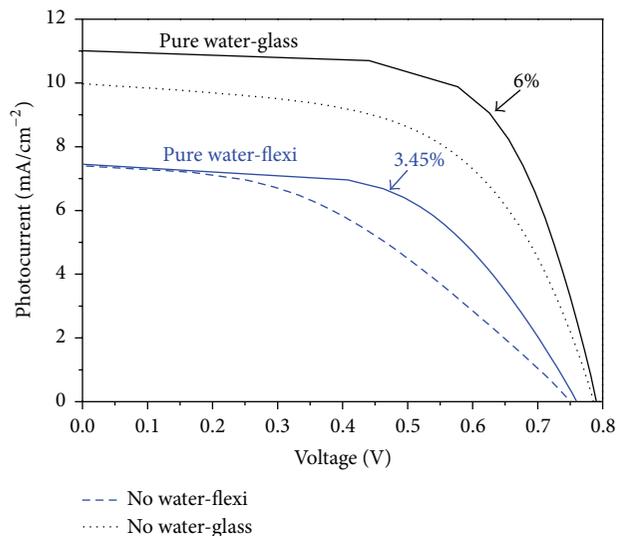


FIGURE 4: I - V curves obtained from a pure water electrolyte for the cell IC100 and standard electrolyte for flexible and glass cells with 1 sun illumination.

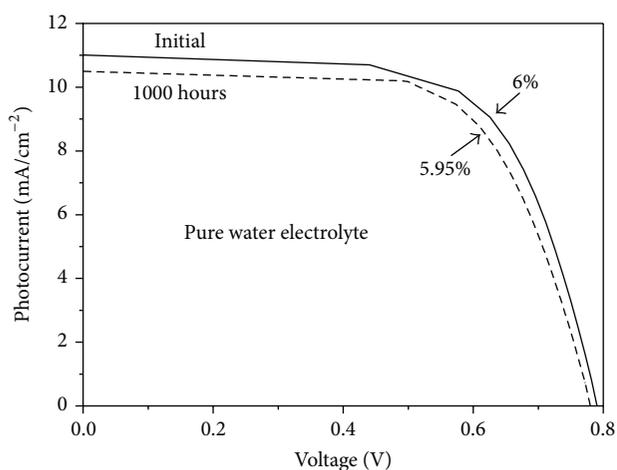


FIGURE 5: I - V evolution of a IC100 under continuous illumination at 1 sun, 60°C.

1.5 G irradiation. Only a 0.8% loss was recorded up to 1000 h under high temperature soaking at 60°C.

4. Conclusion

We prepared and investigated water-based electrolyte glass and flexible DSSCs. The electrolytes containing 100% water show a high solar cell performance of up to 3.45% for flexible cells and 6% for glass cells under simulated AM 1.5 G (100 mW/cm²) irradiation. To the best of our knowledge, this is the highest recorded efficiency until now and good long-term stability under continuous simulated solar light (visible-light irradiation). One of the most important features of using a high concentration of the alkylimidazolium is the reduction in the deleterious effect of water and an improvement in its miscibility with the additives present in

the electrolyte, thus enhancing the solar cell's performance. The role of GuSCN in enhancing the J_{sc} should also be noted. Contrary to previous works highlighting the negative effect of water, our results anticipated that water could be a good candidate for novel designed DSSCs with lower cost and environmentally friendly components.

Conflict of Interests

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

Acknowledgments

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