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Dye-sensitized solar cells using deep eutectic solvents mixed with ethanol as an effective electrolyte medium

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ABSTRACT

This research aims to develop a new type of electrolyte for dye-sensitized solar cells (DSCs) which can be produced in cost-effective and large scale. DSCs using deep eutectic solvents (DESs) mixed with ethanol (50% w/w DES content), as an electrolyte medium, was studied herein for the first time. Ten types of DESs were synthesized and three among them were potential candidates for DSC electrolytes. Compared to toxic and volatile organic solvents, this mixed solvent is more eco-friendly and inexpensive. According to $J-V$ curve measurements, DSCs that used DES-ethanol medium showed promising photovoltaic performance.

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1. Introduction

Dye-sensitized solar cells (DSCs) have attracted widespread scientific and technological attentions as a high efficiency and low-cost alternative to conventional inorganic photovoltaic devices O'Regan and Grätzel (1991); Papageorgiou (1996); Hagfeldt et al. (2010). From the first time of invention by O'Regan and Grätzel in 1991, DSCs have undergone a developmental process in terms of practical applications O'Regan and Grätzel (1991). A typical DSC includes three parts of mesoporous titanium dioxide (TiO_2) film, a photoelectrode, an anchored single layer of dye molecules which are deposited on a conducting substrate (fluorine-doped tin oxide (FTO)), a volatile liquid electrolyte dissolving (I^-/I_3^-) redox couple, and a platinumized FTO glass as the counter electrode Hagfeldt et al. (2010). DSCs have succeeded in utilizing solar energy; however, DSCs currently face several limitations which prevent them from future large-scale applications and commercialization Hagfeldt et al. (2010); Preat et al. (2010).

The electrolyte is an essential component, and its properties play a critical role in the conversion efficiencies and the stability of DSCs Wu et al. (2015); Mohanty and Bhargava (2015). However, one

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of the most obvious drawbacks of conventional DSCs is the usage of the volatile and hazardous organic solvents as electrolyte media Preat *et al.* (2010). Liquid electrolytes also cause significant degradation of dye sensitizers at elevated temperatures Nguyen *et al.* (2009, 2010). Therefore, to overcome these drawbacks new types of solvents, such as ionic liquids (ILs) Lin *et al.* (2016); Decoppet *et al.* (2017); Cao *et al.* (2008); Bidikoudi *et al.* (2014); Li *et al.* (2015), polymers Singh *et al.* (2009), siloxane Lee *et al.* (2013), and deep eutectic solvents (DESs) Denizalti *et al.* (2018); Jhong *et al.* (2009) have been evaluated and applied in DSC fabrication. Among these solvents, DESs stand out as a potential candidate for their availability, inexpensive production and high sustainability, which can allow for large-scale application of this generation of solar cells.

DES is a mixture of two or three cheap and safe components which can connect with each other through hydrogen bond interaction Abbott *et al.* (2004). Generally, DESs are characterized by a vast depression of freezing point and are liquids at temperatures lower than 150°C Abbott *et al.* (2004). Typical DES components, such as urea, glycerol, polyalcohol, carboxylic acids and choline chloride, come from cheap and renewable sources García *et al.* (2015). Thus, DESs are highly biodegradable and inexpensive solvents García-Álvarez (2015); Liu *et al.* (2015); Zhao *et al.* (2015).

DESs share many physicochemical properties with room temperature ILs, such as thermal stability, low vapor pressures, refractive index, conductivity, and surface tension Ruß and König (2012). However, DESs are not composed entirely of ionic species and can also be obtained from nonionic species Ruß and König (2012). Moreover, compared to expensive imidazolium or other organic salts used for the synthesis of ILs, choline chloride (ChCl) which was used to prepared DESs is considerably cheaper. In addition, ILs are toxic, aqueous-sensitive, and non-biodegradable Smith *et al.* (2014).

Applications of DESs in DSCs are still in their infancy. There have been only two reports using DESs as first electrolytes for DSCs with organic dyes, such as D149 Jhong *et al.* (2009) and PTZ-TEG Denizalti *et al.* (2018). Abbotto *et al.* used an aqueous choline chloride-based DES in DSC and achieved photovoltaic performance of the devices fabricated with organic dyes with an overall energy conversion of 0.6% Denizalti *et al.* (2018). Wong *et al.* reported an eutectic mixture of glycerol and choline iodide to apply in DSCs using organic dye, and reached an efficiency of 3.8% Jhong *et al.* (2009).

Therefore, developing new types of DESs is necessary for large scale production of DSCs. In this study, we synthesized ten DESs from choline chloride and different hydrogen bond donors (HBDs). Because the high viscosity of DESs can lead to low ionic mobility or diffusion, we mixed these DESs with ethanol which is abundant and biodegradable (to reduce viscosities as well as to increase the range of solubility of DESs). The appropriate ethanol-DES mixed solvents were used as the electrolyte solvents for their implementation in functional DSCs. Finally, we used *J-V* curve measurements to characterize the cell performances.

2. Experimental Section

2.1 Chemicals and instrumentations

All commercially available reagent-grade chemicals were purchased from Sigma-Aldrich (St. Louis, MO, USA), and used as received without further purification unless otherwise noted.

Proton nuclear magnetic resonance (^1H NMR) and carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker Advance II-500 MHz (Bruker, Rheinstetten, Germany). Fourier-transform infrared (FT-IR) spectra were measured from potassium bromide (KBr) pellets using a Bruker Vertex 70 system (TA Instruments, New Castle, USA). The thermogravimetric analysis (TGA) was recorded on an TGA Q5000 thermal analysis system (TA Instruments, New Castle, USA) with the sample held in a platinum pan with continuous airflow.

2.2 General procedure for the preparation of DESs

The eutectic mixtures of choline chloride were prepared by stirring the two corresponding components at approximately 100°C until a homogeneous, transparent liquid was formed. After completion of the reaction, the reaction mixture was cooled to room temperature. All the products were washed with diethyl ether and then dried under vacuum at 60°C for 6 h before characterization [Zhang et al. \(2012\)](#). The general procedure for the synthesis of DES1 was as follows. DES1 was prepared by mixing choline chloride (5 mM) with 3-phenylpropionic acid (5 mM) at 100°C until a homogenous liquid was formed. The pure product was obtained by simply washing DES1 with diethyl ether and drying under vacuum before use. The other DESs were prepared in a similar way by replacing 3-phenylpropionic acid with other HBDs (**Table 1**). The structure and purity of DES1 were confirmed by FT-IR, TGA, ¹H NMR, and ¹³C NMR.

FT-IR (KBr, cm⁻¹): 3391, 3062, 2963, 1717, 1477, 1080, 955, 701.

¹H NMR (500 MHz, DMSO) δ 7.25 (t, *J* = 7.4 Hz, 2H), 7.21 – 7.12 (m, 3H), 3.84 (m, 2H), 3.54 – 3.42 (m, 2H), 3.17 (s, 9H), 2.79 (t, *J* = 7.6 Hz, 2H), 2.52 (t, *J* = 7.6 Hz, 2H).

¹³C NMR (125 MHz, DMSO) δ 174.0, 141.1, 128.5, 128.3, 126.2, 67.1, 55.3, 53.5, 35.5, 30.5.

2.3 DSC fabrication and characterization

DSCs were prepared per previous studies [Decoppet et al. \(2017\)](#); [Boldrini et al. \(2017\)](#); [Šurca Vuk et al. \(2008\)](#).

2.3.1 Preparation of photoanodes

FTO glass (Pilkington, USA; 8Ωcm⁻²) with size of 2×2 cm was ultrasonically cleaned with a detergent solution, distilled water and ethanol, respectively. Subsequently, the glass was dried and treated with 40 mM aqueous solution of titanium tetrachloride (TiCl₄) at 70°C for 30 min and then rinsed with water and ethanol. A transparent layer of 0.25 cm² was screen-printed using TiO₂ paste (Dyesol 18NR-T). The coated transparent film was dried at 100°C for 10 min, and another layer was screen-printed by using a light scattering TiO₂ paste (Solaronix R/SP). The coated films were thermally treated at 500°C for 30 min. After cooling down to room temperature, the TiO₂ coated plate was immersed in a 0.42 mM solution of the N-719 dye for 20 h at room temperature in the dark.

2.3.2 Preparation of the counter electrodes

Two 1-mm holes were drilled in a FTO plate (1.5×2 cm), using diamond drill bits. The electrodes were then cleaned with a detergent solution for 30 min using an ultrasonic bath. The platinum (Pt) (Solaronix, Switzerland) counter electrode was coated on FTO template. The coated transparent film was then dried at 500°C for 30 min.

2.3.3 Assembly of cells

The photoelectrode was assembled with the Pt counter electrode to form a sandwich-type cell. Then, the electrolyte was injected into the cell. Finally, the holes were sealed with a sheet of surlyn and a cover glass. The electrolyte consisted of 0.3 M *tetrabutylammonium iodide* (TBAI), 0.05 M iodine (I₂) and 0.25 M 4-*tert*-butylpyridine (4-*t*BP) dissolved in DES-ethanol mixed solvent (50% w/w DES content).

2.3.4 DSC measurements

Photovoltaic measurements of DSCs were performed according to a previously published protocol [Tran et al. \(2017\)](#); the DSCs were masked with a 0.144 cm² active area of the anode electrode.

3. Results and Discussion

3.1 The preparation of DESs

We generated DESs by mixing quaternary ammonium salts (choline chloride) with HBDs in a suitable molar ratio. The deep eutectic solvents in the electrolytes were prepared from choline chloride and varied HBDs, such as 3-phenylpropionic acid, succinic acid, ethylene glycol, oxalic acid, glucose, fructose, urea, pyrrole, imidazole, and indole. Thus, they were identified as DES1, DES2, DES3, DES4, DES5, DES6, DES7, DES8, DES9, and DES10, respectively. The yields of conversions are listed in **Table 1**.

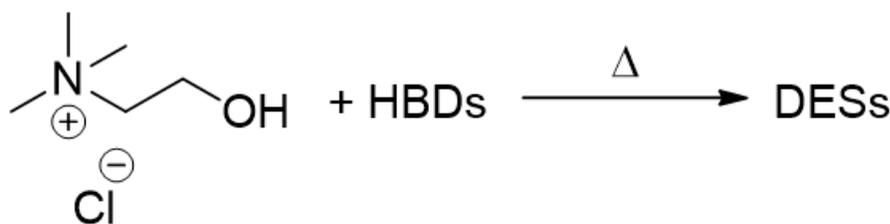


Table 1. The preparations of DESs from choline chloride and HBDs at 100°C

Sample	HBDs	Mole ratio (ChCl:HBD)	State	Time (min)	Yield (%)
DES1	3-phenylpropionic acid	1:1	L	30	95
DES2	Succinic acid	1:1		30	97
DES3	Ethylene glycol	1:1	L	20	98
DES4	Oxalic acid	1:1	L	30	98
DES5	Glucose	1:1	L	30	~100
DES6	Fructose	1:1	L	30	~100
DES7	Urea	1:2	L	30	98
DES8	Pyrrole	3:7	L	10	~100
DES9	Imidazole	3:7		25	95
DES10	Indole	3:7		25	96

S: solid, L: liquid

These DESs were synthesized to meet several main requirements: available raw materials, fast and low-cost preparation, sustainability, a wide range of solubility, and a complete absence of metal ions which may affect DSC performance.

3.2 Solubility test

To be applied as solvents in the liquid electrolyte preparation in DSCs, DESs should be inert with the surface-attached dye N719. In addition, DESs must be soluble in an ethanol solution containing the redox couple (I^-/I_3^-) and the additive 4-*t*. The results are shown in **Table 2**. Since the DESs showed high

viscosities, they were dissolved in ethanol which is an abundant and inexpensive solvent to reduce their viscosities as well as enhance their solubilities.

Table 2. The solubility test of conventional solvent and synthesized DESs

Solvent	Dye N-719	Ethanol (, 4-t)	Side-reaction(*)
DES1	I		-
DES2	I		-
DES3	I		-
DES4			-
DES5	I	I	-
DES6	I	I	-
DES7	I	I	-
DES8	I	I	Reaction with I ₂
DES9	I	I	Reaction with I ₂
DES10	I	I	Reaction with I ₂

I: insoluble, S: soluble. (*): reactions between DES and components in electrolyte or materials which were used to prepare the cell.

Ethanol-based DES1 to DES4, synthesized from choline chloride and HDBs (3-phenylpropionic acid, succinic acid, oxalic acid and ethylene glycol), turned out to be potential solvents for reparation of electrolyte in DSCs. Although DES4 can be soluble in ethanol containing the redox couple and additives, DES4-made Dye N719 desorbs from the TiO₂ surface. Therefore, DES4 (choline chloride-oxalic acid) cannot be used in DSCs. Moreover, DES4 to DES10 are insoluble in ethanol, which means they could not be used as ethanol-DES electrolytes in DSCs. In addition, DES8 to DES10 readily react with I₂ and decompose; therefore, they cannot be used.

3.3 Thermal gravimetric analyses (TGA)

TGA of DES1, DES2, and DES3 were investigated to test their thermal stability. The results are shown in **Figure 1**. The analysis shows that three DESs are stable at high temperatures (up to 150°C), which confirms these three DESs as suitable for serving as electrolytes in DSCs.

The three DESs (DES1 to 3) mixed with ethanol were then implemented in DSCs as the electrolyte solvents. The results of the photovoltaic performance of DSC using DES1, DES2 and DES3 are summarized in **Table 3**, and the corresponding *J-V* curves are illustrated in **Figure 2**.

Table 3. Photovoltaic performance of DSCs fabricated with DES1-3 in electrolyte

Cell	DES mixed with ethanol*	J _{sc} (mA.cm ⁻²)	V _{oc} (V)	Fill Factor	Efficiency (%)
1	DES1	5.07	0.72	0.47	1.7
2	DES2	4.37	0.69	0.52	1.6
3	DES3	4.53	0.76	0.57	2.0

* Ethanol:DES (50% w/w DES content)

DSCs were measured at 1 sun. The results were the average of several cell

As can be seen from **Table 3** and **Figure 2**, cells using an electrolyte with the three different DESs showed good photovoltaic performance, according to the shape of the *J-V* curves. The open circuit voltage

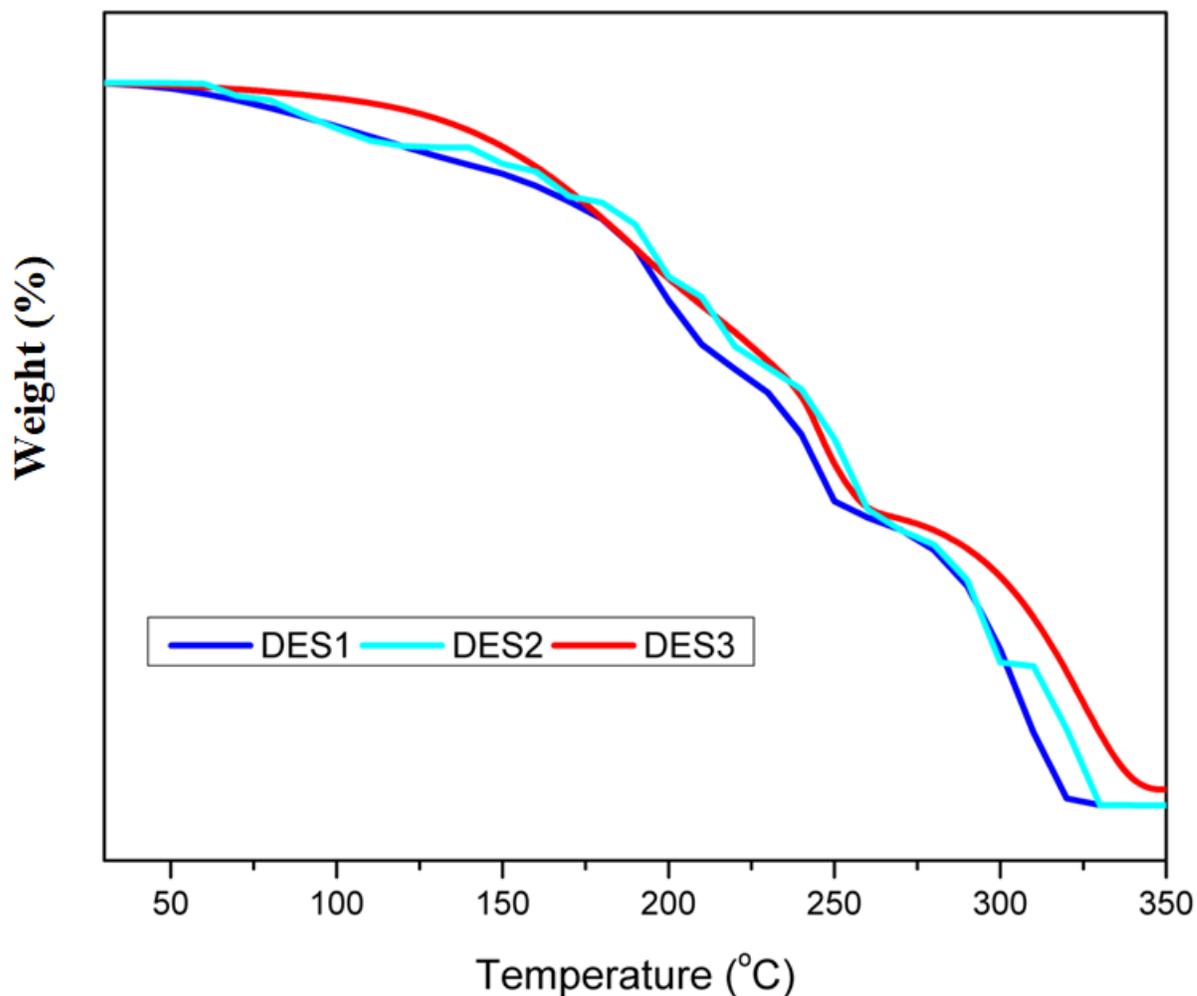


Figure 1. Thermogravimetric analysis (TGA) curves of DES1, DES2, and DES3 performed on a TA Q500 thermal analysis system with the sample held in a platinum pan with a continuous airflow.

(V_{oc}) of all these cells showed significantly high values (> 0.7 V), whereas the value was reported to be around 0.55 V in the same electrolyte condition using the redox couple I^-/I_3^- [Jhong et al. \(2009\)](#). However, the short circuit current density (J_{sc}) values were rather low. This situation may occur due to lack of optimized fabrication process in applying the DESs to DSCs.

The cells using ethanol mixed with DES3 (choline chloride -ethylene glycol as the electrolyte media) showed the best performance and reached a conversion efficiency of 2.0%. In addition, its open-circuit voltage and fill factor (FF) were also the highest among the cells. This can be explained by the lowest viscosity of 37 cP (25°C) [Tang et al. \(2015\)](#) and the highest conductivity of $7.61 \text{ mS}\cdot\text{cm}^{-1}$ (25°C) [Tang et al. \(2015\)](#) for DES, compared to the two other DESs. It is obvious in **Figure 2** that DSCs using DES2 gives the worst performance which has a good agreement with its highest viscosity (8103 cP, 25°C) [Zhang et al. \(2012\)](#) and low conductivity ($0.31 \text{ mS}\cdot\text{cm}^{-1}$, 25 °C) [Zhang et al. \(2012\)](#), leading to low mobility and diffusion of the mediator species in the electrolyte mixture. Even though the overall energy conversion efficiencies of DSCs using DES1 and DES2 are not much different, the shape of their J-V curves and the three photovoltaic parameters show noticeably dissimilar behavior. The shape of J-V curve of the cells with DES1 indicates a reduction of shunt resistance leading to low fill factor. This might be explained by the

fact that DES1 showed the lowest conductivity of $0.16 \text{ mS}\cdot\text{cm}^{-1}$, 25°C Zhang et al. (2012). Nevertheless, the high current and high voltage of DSCs using DES1 were attained, in contrast to the fill factor, probably since its viscosity (365 cP) is less than half of that of DES2 Zhang et al. (2012).

To the best of our knowledge, this is the first time that ethanol-based DESs have been applied in DSCs. Regarding the electrolyte components, we preferred to limit the investigation to widely available iodine-based chemicals as our main scope to test the ethanol-DES solvent. In this work, the electrolyte consisted of 0.3 M TBAI, 0.05 M I_2 and 0.25 M 4-*t* dissolved in ethanol dissolving DES (50% w/w DES content). Therefore, the conditions have not been optimized yet to achieve the best photovoltaic performance. Besides electrolyte composition, other parameters (including dye concentration and TiO_2 thickness) should be optimized to improve cell performance. We believe ethanol-based DESs can be considered as a potential electrolyte solvent that can be implemented in DSCs.

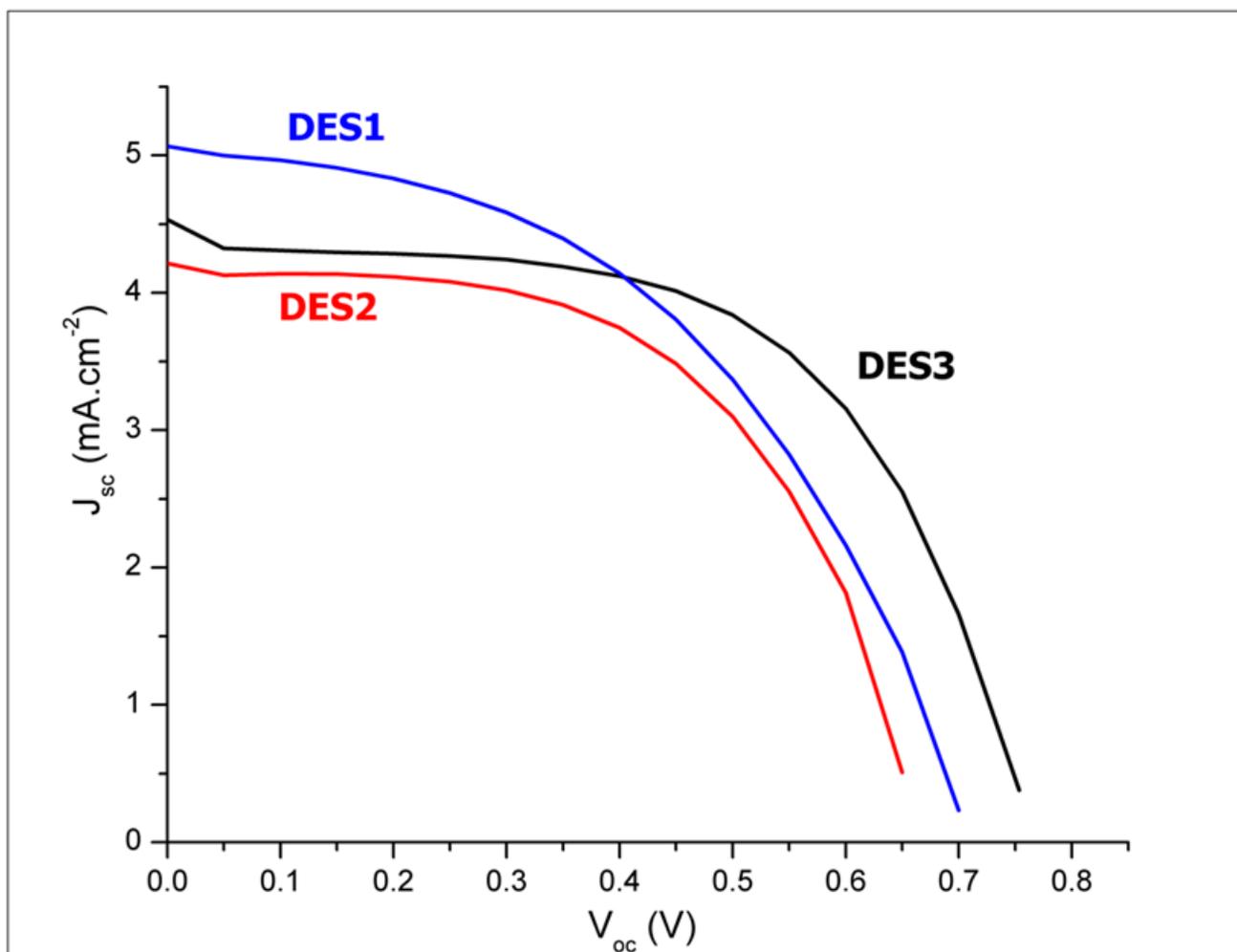


Figure 2. The J-V curves of DSCs using ethanol-DES mixed solvents.

4. Conclusion

In summary, ten different DESs were successfully prepared from choline chloride and HBDs by a simple process. Among these, three DESs made from choline chloride and HBDs (3-phenylpropionic acid,

succinic acid, and ethylene glycol) have turned out to be potential DES-ethanol mixed solvents for the preparation of electrolytes in DSCs. Compared to conventional solvents, the DES-ethanol mixed solvents used as electrolyte media showed high open-circuit voltages (> 0.7 V), fill factors (~ 0.55), and good density currents (ranging from 4 to 5 mA.cm⁻²). However, the cell fabrication in this study was not optimized; therefore, there is still room to improve on the DSC efficiency. Moreover, these DESs showed several benefits, such as low-cost production, easy preparation, and environmentally friendly solvents, which allow DESs to be a strong candidate for DSC electrolyte development to large scale production.

5. Acknowledgment

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6. Author Contributions

Phuong Hoang Tran and Phuong Tuyet Nguyen designed and performed the experiments; Thuy-Duy Thi Nguyen and Phuong Tuyet Nguyen analyzed the spectra and data; all authors wrote the paper and approved the final manuscript.

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