



Three-dimensional network electrolytes with highly efficient ion-transporting channels for quasi-solid-state dye-sensitized solar cells



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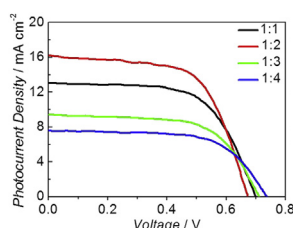
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HIGHLIGHTS

- Three-dimensional network polymers (PAAPEs) are synthesized.
- This structure provides efficient ion-transporting channels for gel electrolyte.
- The optimized gel electrolyte shows a conductivity of 4.03 mS cm^{-1} at 25°C .
- The DSC based on PAAPE electrolyte obtains a η of 6.81%.

GRAPHICAL ABSTRACT



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ABSTRACT

Quasi-solid-state dye-sensitized solar cells (DSCs) are fabricated with three-dimensional network poly(adipic acid pentaerythritol ester) (PAAPE). The PAAPEs are prepared by esterification of pentaerythritol and adipic acid. This three-dimensional structure provides highly efficient ion-transporting channels for iodide/triiodide (I^-/I_3^-) transport in the gel electrolyte. The optimized gel electrolyte shows conductivity of 4.03 mS cm^{-1} at 25°C . The fabricated DSC obtains a photoenergy conversion efficiency of 6.81% under AM 1.5 irradiation.

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1. Introduction

Dye-sensitized solar cells (DSCs) have attracted great attention for the past decade because of high photon-to-current conversion efficiency and low production cost [1,2]. DSCs usually comprise an electrode consisting of a nanocrystalline titanium dioxide (TiO_2) film modified with a dye, a platinum counter electrode and an

electrolyte between the electrodes. Photoexcitation of the dye results in the injection of an electron into the conduction band of the oxide. The original state of the dye is subsequently restored by electron donation from a redox system, such as the I^-/I_3^- couple. Currently, DSCs are mainly constructed by using liquid electrolytes as charge transport material, because liquid electrolytes provide free volume for the typical charge transport. Recently, a solar-to-electricity conversion efficiency of 12.3% has been achieved by employing a liquid electrolyte containing cobalt based redox shuttles [3]. However, liquid electrolytes have disadvantages such

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as evaporation and leakage of the solvents under practical long-term operation.

In order to solve these problems, several attempts were made to replace the liquid electrolytes with less volatile electrolytes such as *p*-type semiconductors [4–8], room temperature ionic liquids [9–11], hole transporting materials [12,13], addition compounds [14,15], polymer gel electrolytes [16–23] and so on. Among these, polymer gel electrolytes were especially interesting due to their diversity and ease of modification. Most polymer gel electrolytes were prepared by incorporating liquid electrolytes directly into polymer matrices, such as poly(ethylene glycol) [24], poly(1-butyl-3-vinylimidazolium bis(trifluoromethanesulfonyl) imide) ([PBVIm][TFSI])[23], polyacrylonitrile/grapheme [25]. In general, the structures of the commonly used polymer gel electrolytes were one- or two-dimensional and the polymer chains could hinder ion conducting in these gel electrolytes. As a result, the conductivity (σ) of them was poor, and then lowering conversion efficiency of DSCs. For instance, the conductivities of above-mentioned electrolytes were 0.05–3 mS cm⁻¹, and the efficiencies of corresponding DSCs were 3–5%, respectively [24,25]. In more recently, several new polymer electrolyte systems were reported, for example, two-dimensional graphene and soluble polystyrene nanobead based composite electrolyte [26], poly(oxyethylene) diamine (POE-amine) and an aromatic anhydride copolymer based electrolyte [27], and polypyrrole integrated poly(hydroxyethyl methacrylate/cetyltrimethylammonium bromide) based electrolyte [28], etc. However, the structures of the polymers were still one- or two-dimensional.

If we can synthesize three-dimensional network polymer, the electrolyte prepared by this polymer will have highly efficient ion-transporting channels. Thus, high conductivity of electrolyte as well as high conversion efficiency of DSCs can be expected. Keeping this in mind, we employ poly(adipic acid pentaerythritol ester) (PAAPE) to prepare three-dimensional network structure electrolyte. By crosslinking reaction, a three-dimensional network polymer is formed, where the building blocks are connected through ester groups. The three-dimensional structure of the polymer matrix can absorb liquid electrolyte and provide ion-transporting channels.

2. Experimental

2.1. Materials

All reagents used were analytical grade. Pentaerythritol (Sinopharm Chemical Reagent Co., Ltd.), adipic acid (Sinopharm Chemical Reagent Co., Ltd.), *p*-toluenesulfonic acid (Sinopharm Chemical Reagent Co., Ltd.), I₂ (Sinopharm Chemical Reagent Co., Ltd.), and KI (Sinopharm Chemical Reagent Co., Ltd.) were used as received. Propylene carbonate (PC) and ethylene carbonate (EC) were supplied by Aladdin. TiO₂ (P25, Degussa AG of Germany) consists of 30 wt% rutile and 70 wt% anatase. The conductive glass of fluorine-doped SnO₂ (FTO, 14 Ω/sq) was purchased from Asahi Glass. Di-tetrabutylammonium cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II) (N719) was supplied by Yingkou OPV Tech New Energy Co., Ltd. Other reagents were purchased from Shanghai No.4 Reagent & H.v Chemical Reagents Company.

2.2. Preparation of PAAPE

PAAPE[1:1], PAAPE[1:2], PAAPE[1:3] and PAAPE[1:4] were respectively synthesized by pentaerythritol and adipic acid with molar ratios of 1:1, 1:2, 1:3, 1:4, respectively. Pentaerythritol was dissolved in the fusing adipic acid and 5 wt% *p*-toluenesulfonic acid was added as catalyst at 170 °C for 1 h. The above mixture was degassed under vacuum for eliminating water. Its reaction formula

and three-dimensional network structure schematic drawing are illustrated in Fig. 1a. Fig. 1b–e are the pictures of PAAPEs. PAAPE[1:1] is a tough and glutinous polymer and PAAPE[1:2] is tough without viscosity. PAAPE[1:3] and PAAPE[1:4] are fragile. Fig. 1f–i are the pictures of PAAPE-based electrolytes. It should be noted that *p*-toluenesulfonic acid as a catalyst still presents in the as-prepared PAAPEs. But its content (only 5%) is too low to be detected by FTIR. Likewise, its influence on the electrolyte can also be ignored.

2.3. Preparation of electrolytes

The polymer gel electrolytes were obtained by immersing the PAAPEs into the I⁻/I₃⁻-based liquid electrolyte at 80 °C for 24 h. The liquid electrolyte contained 0.6 M KI and 0.06 M I₂ in PC/EC (1/4) binary solvent.

2.4. Fabrication of the cells

The 8 μm-thick nanocrystalline TiO₂ film was fabricated on a conducting glass substrate by a screen printing technique [29]. The TiO₂ film was sintered at 450 °C for 30 min [30]. After cooling down to 80 °C, it was immersed in an acetonitrile solution containing 5 × 10⁻⁴ M of ruthenium dye N719 overnight to adsorb dye molecules. The DSC was made by sandwiching the electrolyte between the dye-covered TiO₂ electrode and the Pt counter electrode under pressure [31].

2.5. Measurements

Infrared absorption spectra were recorded on a Fourier transform infrared (FTIR) spectrometer (Thermo Fisher Scientific: Nicolet 6700). Scanning electron microscope (SEM) images were obtained using a scanning electron microscope (HITACHI: E-1010). Photoelectrochemical measurements were performed according to the reported procedures [32]. Photoenergy conversion efficiency was evaluated using a solar simulator (ABET: Sun 3000). Thermogravimetric analysis (TGA) was tested employing simultaneous thermal analyzer (STA409C/PC).

Electrochemical impedance spectroscopy (EIS) of the cells was measured using electrochemical workstation (Autolab: PGSTAT302N). The temperature of the electrolytes was controlled at 25 °C. The conductivity of the gel electrolyte was determined by impedance measurement. The electrolyte was sandwiched between two mirror-finished stainless steel electrodes using a Teflon ring spacer in a constant volume cylindrical cell. The conductivity was calculated from the ionic resistance (*R*), which was obtained from the intercept on the real part of the impedance in the complex plane, by the following equation $\sigma = K_{\text{cell}}/R$ [33]. *K*_{cell} is cell constant tested by using standard potassium chloride solution.

3. Results and discussion

3.1. Characterization of PAAPE

Fig. 1a illustrates the reaction formula and three-dimensional network structure schematic drawing of PAAPEs. Pentaerythritol, as a core in the PAAPEs, has a three dimensional structure, in which it has 4 hydroxyl in 4 directions. The three dimensional pentaerythritol is connected by adipic acid through esterification reaction. So the PAAPEs theoretically have a three dimensional structure, though some irregular structure maybe exist in PAAPEs.

FTIR spectra are used to investigate the four PAAPEs. Due to the different stoichiometry, PAAPE[1:1] contains functional groups of –OH and –COOC–, PAAPE[1:3] and PAAPE[1:4] have functional groups of –COOH and –COOC–. However, PAAPE[1:2] only has

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