



Preparation of highly concentrated and stable conducting polymer solutions and their application in high-efficiency dye-sensitized solar cell



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ABSTRACT

Highly concentrated, good dispersed and stable conducting polyaniline and poly(3,4-ethylenedioxythiophene)/hexafluoro-isopropanol (HFIP) colloid solutions (PANI/HFIP and PEDOT)/HFIP) are prepared simply by dissolving the doped polymer powders in HFIP. The hydrogen bonding between the doped polymer and fluorinated solvent is not only a driving force for forming concentrated and stable solution but also disintegrates polymer chains in the colloid particles to form more expanded coil-like conformation. PANI-SO₄-F (H₂SO₄ doped PANI) and PEDOT-F (doped PEDOT) films casted from the corresponding HFIP solutions have not only high electrical conductivity and electrocatalytic activity but also high surface area and good adhesion on the substrate. Dye-sensitized solar cells (DSCs) using CYC-B11 sensitizer, I⁻/I₃⁻ redox pair electrolyte and PANI-SO₄-F or PEDOT-F/FTO counter electrode achieve the conversion efficiency (η) of 8.8–9.0% which is as good as those for DSCs based on Pt/FTO counter electrode fabricated with the same procedure using the same sensitizer and electrolyte. The excellent photoelectric properties, easy preparation and low cost make conjugated polymer to be a credible material for replacing Pt in DSCs.

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1. Introduction

Mesoscopic dye-sensitized solar cells (DSCs) have emerged as one of the most promising candidates for the practical photovoltaic applications in virtue of their low manufacturing cost and impressive conversion efficiency [1–3]. In DSC, a platinum film was coated on the counter electrode to catalyze the reduction (I₃⁻ to 3I⁻) of the redox electrolyte to keep the low overvoltage at a reasonable photocurrent density [4–6] and to accelerate the redox reaction [7–10]. It has been estimated [11] that using conducting polymers instead of Pt as a catalyst of the counter electrode can reduce 30–40% cost of the whole

photovoltaic cell regardless of the preparation methods. Therefore many studies focused on using conjugated polymers (amongst other carbon based materials) such as poly(3,4-ethylenedioxythiophene) (PEDOT) [12], polypyrrole (PPy) [13] or polyaniline (PANI) [14] to replace Pt in the counter electrode of a DSC. Up to now the highest conversion efficiencies up to 7.93%, 7.66% and 7.36%, for PEDOT, PPy, and PANI based devices, respectively have been achieved. However, these conjugated polymer counter electrodes were prepared either *via* electrochemical or *in situ* polymerization method or used very complicated procedure, which increases the fabrication cost. Therefore the fabrication process and performance of the polymer counter electrode need to be improved to really reduce the cost of DSCs.

On the other hand, fluorinated alcohol such as hexafluoro-isopropanol (HFIP, a non-toxic liquid with low

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boiling point (59 °C)) with mildly acidic and strong hydrogen-bonding capability was known to be a good solvent for organo-sulfonic acid doped emeraldine salt. ES-PANI film made from HFIP solution thus has an ordered structure and good conductivity as revealed with absorption spectrum, X-ray diffraction study, and conductivity measurement [15,16]. In this article, we used HFIP as a solvent for the doped polyaniline (PANI) and poly(3,4-ethylenedioxythiophene) (PEDOT) to prepare PANI/HFIP and PEDOT/HFIP colloid solutions. DSCs used PAIN-F and PEDOT-F (films fabricated by spin coating or casting from PANI/HFIP and PEDOT/HFIP solutions at room temperature) counter electrode combined with CYC-B11 sensitizer [17] achieves the overall conversion efficiency of 8.8% and 8.9%, respectively. The efficiencies are the highest for DSCs based on polymer counter electrodes reported in the literature and are as good as that for the device based on Pt counter electrode fabricated at the same procedures.

2. Experimental section

2.1. Chemicals

Water was purified using a Milli-Q (Millipore Corp., Bedford, MA) purification system with a resistivity of 18 M Ω cm. Aniline and ethylenedioxythiophene (ACROS, ACS grade) were used after distilling over calcium hydride and stored in the dark under nitrogen atmosphere. Hexafluoro-isopropanol (HFIP), acetone, chloroform, toluene, HCl_(aq), N-methyl pyrrolidinone (NMP), MeOH, 0.1 M NH₄OH_(aq), H₂SO_{4(aq)}, phenol, and (NH₄)₂S₂O₈ were obtained from the commercial resources and used without further purification. Polyaniline powders (with HCl or H₂SO₄ as a dopant) were prepared according to our previous report [18].

2.2. Preparation of poly-ethylenedioxythiophene via chemical polymerization method

Dissolving 0.5 g ethylenedioxythiophene (EDOT) in 50 ml 1.9 M HCl_(aq) to make 0.07 M EDOT_(aq) (solution (a)). 0.82 g of [(NH₄)₂S₂O₈] was dissolved in 1.0 M, 20 ml HCl_(aq) to make 0.18 M [(NH₄)₂S₂O₈]_(aq) (solution (b)). Solution (b) was added slowly to solution (a) and the mixture was stirred at room temperature for 24 h. Polymer powder was filtrated and washed sequentially with large amount of H₂O, 0.1 M HCl_(aq), MeOH, acetone, and acetonitrile, then dried in a vacuum.

2.3. Preparation of conducting polymer solutions and the solubility measurements

Doped conjugated polymer powders were added in HFIP gradually to make an over saturated polymer colloid solution. The colloid solution was sonicated for 10 h at room temperature and then passed through a 20 μ m filter to remove the un-dissolved particles. The resulting polymer colloid solutions were named as PANI-Cl/HFIP and PEDOT/HFIP. To demonstrate the merit of using HFIP as a solvent for conjugated polymers, HCl_(aq) (pH = 2.6), NMP,

and MeOH were also used as solvents to dissolved HCl doped polyaniline powder to form PANI-Cl/HCl_(aq), PANI-Cl/NMP and PANI-Cl/MeOH solutions, respectively. Conducting polymer films named as PANI-Cl-F, PEDOT-F, PANI-Cl-A, PANI-Cl-N, and PANI-Cl-M were made from the corresponding PANI-Cl/HFIP, PEDOT/HFIP, PANI-Cl/HCl_(aq), PANI-Cl/NMP and PANI-Cl/MeOH solutions, respectively. The solubility of doped polymer in a solvent was determined as the following: the accurate volume of the polymer solution was carefully transferred into a weighted vessel. Solvent in the polymer solution was removed by vacuum drying and the weight of the polymer with vessel was measured to calculate the net weight of the polymer and then the concentration of the polymer colloid solution.

2.4. Fabrication of DSCs with conducting polymer coated FTO counter electrode and their photovoltaic performance measurements

Doped polymer film was deposited on the surface of the cleaned fluorine-doped tin oxide glass (FTO, sheet resistivity of 15 Ω /square) by spin-coating method. The procedure for fabricating DSCs is the same as what we reported previously [19], except a polymer film (as well as thermal Pt)-coated FTO plate was used as a counter electrode. The characteristic current density–voltage (*J*–*V*) of the cell under the illumination of AM 1.5G simulated sunlight (Yamashita Denso Corporation, YSS-50A, 100 mW cm⁻²) were obtained by applying external potential bias to the cell and measuring the photocurrent output with a Keithley model 2400 digital source meter (Keithley, USA).

2.5. Physicochemical studies

UV/Vis/NIR spectra were obtained using Varian Cary 5E spectrophotometer in the laboratory atmosphere at room temperature. Scanning Electron Micrograph (SEM) was recorded with a Hitachi S-800 at 15 kV. The samples (film on substrate) for SEM imaging were mounted on the metal stubs with a piece of conducting tape and then coated with a thin layer of gold film to avoid charging. TEM SAED (selective area electron diffraction) patterns were taken from a JEOL JEM-2000FX. Samples for TEM study were prepared by dipping a drop of polymer solution in a carbon coated grid, dried under vacuum before TEM viewing. X-ray photoelectron spectroscopy studies were carried out on a Perkin-Elmer PHI-590AM XPS/ESCA spectrometer system with a Cylindrical Mirror Electron (CMA) energy analyzer. The X-ray sources were Al K α at 600 W and Mg K α at 400 W. Samples for C-AFM studies were mounted, via silver paste, to AFM sample holder. The contact mode AFM with a current-sensing module (SPA400, SEIKO Inc., Japan) was used to simultaneously obtain the morphology and current image of PANI film on FTO. The viscosity measurements were performed with a viscometer (LVDV-II+PCPE-40) from Brookfield Engineering Laboratories, Inc. The dynamic light scattering (DLS) and zeta potential measurements were carried out with the Zetasizer Nano from Malvern Instruments Ltd. Electrochemistry was performed in a single-compartment, three-electrode cell with a Pt coil counter electrode and an Ag/AgCl reference

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