



Electropolymerized polythiophene photoelectrodes for photocatalytic water splitting and hydrogen production

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ABSTRACT

We present thiophene-based devices fabricated via spin-coating and electropolymerization (EP) for usage in solar-powered, photocatalytic hydrogen gas (H_2) harvesting. Two innovative claims are achieved in this work: (1) demonstration of electropolymerized photoelectrochemical (PEC) devices for water splitting, and (2) drastically improved performance of EP-PEC devices over spin-coated PEC hydrogen harvesters, achieving >0.5 V improvement in onset voltage (V_{on} , bias voltage needed to produce photocurrent), with V_{on} of 0 V vs. Ag/AgCl. As such, this work points to new opportunities for material and device fabrication for cheaper and efficient PEC hydrogen-harvesting systems.

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1. Introduction

As a zero-emission liquid fuel for fuel cells, hydrogen gas (H_2) may soon be a competitive replacement for fossil fuels in the US transportation sector [1]. In addition, H_2 is already widely used in industry applications: for example, as a reactant for metal finishing, as a precursor to chemical processes such as the Haber–Bosch process, and even as a high-energy density fuel in aerospace applications. However, current methods of H_2 production rely heavily on fossil fuels. The most widely used method today, steam reformation ($CH_4 + 2H_2O \rightarrow CO_2 + 4H_2$), not only produces carbon emissions in the H_2 production process itself but also requires maintaining high temperatures (700–1100 °C) and is highly energy intensive [2]. For this reason, more efficient, less energy-intensive, less carbon-intensive hydrogen production methods are needed.

In contrast, photoelectrochemical (PEC) water splitting devices, working ideally, will passively (without bias voltage) convert sunlight into electron/hole pairs that will oxidize water ($2H_2O + 4h^+ \rightarrow 4H^+ + O_2$) and reduce resulting H^+ ions ($4e^- + 4H^+ \rightarrow 2H_2$). As opposed to that of a photovoltaic (PV) cell, a PEC water splitting semiconducting electrode uses generated photocurrent to catalyze red/ox reactions in aqueous medium [3]. Since water splitting is easily scalable with the area of semiconducting material used, it

is ideal for large scale hydrogen harvesting. In contrast, achieving large-scale hydrolysis using PVs requires multiple cells to be stacked in series, increasing resistivity across the H_2 production system, and decreasing conversion efficiency [4].

However, developing materials for efficient conversion of solar energy into fuel has been difficult to accomplish. Ideal water splitting materials require a specific band structure and excellent photocatalytic properties. The band structure should exhibit the following characteristics: (1) band gap (E_g) must be small to absorb the solar spectrum effectively; (2) E_g must be positioned correctly to allow for the red/ox reactions necessary to split water (conduction band above oxidation potential to donate electrons to the oxidation reaction and valence band below the reduction potential); and (3) materials must exhibit high charge separation efficiency in contact with an aqueous electrolyte. Moreover, materials often exhibit poor V_{on} (require large bias voltages to observe photocurrent) due to poor conductivity, recombination, and undesirable band structure [5].

Recent approaches to increasing photocurrent and decreasing onset voltage attempt to increase conversion efficiency, absorbing more of the solar spectrum by using dye-sensitizers or using catalysts to lower overpotential needed to split water [6]. Some organic dye-sensitizers enhance performance by increasing light absorption; the bulk material provides a stable charge-transport base for the moisture-sensitive organic material [7–9]. The addition of hydrogen evolution or oxygen evolution catalysts to water splitting materials is also crucial to increasing photocurrent and

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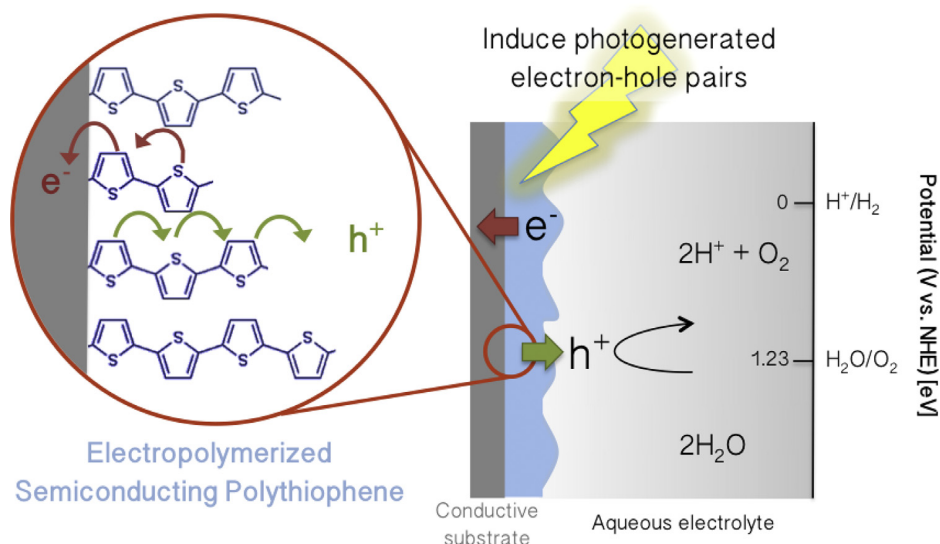


Fig. 1. Representation of surface reactions at electropolymerized polythiophene (and counter electrode) to allow for hydrogen production. A texturized, electropolymerized, semiconducting polymer allows for water oxidation at the surface and hydrogen reduction at an opposite counter electrode.

decreasing onset voltage. Indeed, some semiconducting materials show little observable photocurrent without a catalyst. Only recently have devices with organic material as the bulk material (as opposed to dye-sensitized) been realized as effective water splitting photoelectrodes [10].

Conductive polymers have been found to exhibit characteristics favorable for many applications, such as gas sensors, LEDs, FETs, or photovoltaics [11,12]. Moreover, due to conductive polymers' tunable optical properties in organic electronics, efforts toward cheaper, more efficient, and more robust pathways to polymerization were developed (such as Ziegler–Natta, electrodeposition, Negishi, and Suzuki [13]). Of these, electrodeposition, in which monomer-rich electrolyte solutions undergo polymerization under electric current at the substrate–electrolyte interface, has been shown to achieve rapid, robust, conformal deposition over a highly texturized, highly porous substrates [14,15].

Although cheaper, thiophene-based materials, such as poly-3hexylthiophene (P3HT), have been used widely in photovoltaic applications [16,17] and in organic electronic or optoelectronic devices [18], P3HT proves difficult to use as a water splitting material because of its susceptibility to moisture. Previous works utilizing thiophene-based polymers attempt to stabilize the material with oxide coatings such as titanium dioxide. However, this presents a trade-off between stability and performance. Thicker TiO₂ passivation layers allow for more stable hydrogen generation (>3 h) but also limit solar absorption and decrease conductivity, lowering photocurrent. Despite recent advances in polymerization techniques, existing works on bulk organic materials for water splitting focus on various methods of spin-coating [7]. However, these methods significantly limit the potential of sophisticated fabrication of devices. In particular, texturization of substrates has been shown to increase active area, increase solar absorption, and improve charge separation [3]. By limiting device architectures to planar substrates, existing methods such as spin-coating reduce efficacy of organic substrates in relation to well-known metal oxide semiconductors, of which studies in texturization are numerous [19,20].

This work presents proof-of-concept demonstrations of electroplated polymer materials for photocatalytic water splitting [21]. By using polythiophene (PT), we are able to produce an n-type, thiophene-based PEC device. By electropolymerizing PT, we dope it

in situ, allowing for hole and electron transport into the electrolyte and to the counter electrode, respectively (Fig. 1).

1.1. Electrode design

Fig. 2a shows a conceptual realization of the water splitting system. When the system is illuminated, in an n-type semiconductor, charge separation occurs, band bending allows holes to enter into the electrolyte, and electrons flow into the counter electrode. Furthermore, since electron affinity of polythiophene allows its conduction and valence bands to straddle the red/ox potentials of the water splitting system, PT is a good candidate material for water splitting. The detailed band diagram of the tin electrical contact, PT, and electrolyte are shown in Fig. 2b. Thus, electroplated PT, with favorable band structure for both solar absorption and photocatalysis is able to achieve $V_{on} = 0$ V vs. Ag/AgCl, even without the use of a surface oxygen- or hydrogen-evolving catalyst.

For this paper, we utilize spin-coated P3HT devices as reference devices for comparison purposes. Spin-coated P3HT was chosen for its popularity in the literature, and its comparable characteristics when compared to PT. While both are thiophene-based, they also exhibit comparable bandgaps in the visible range.

1.2. Cell efficiency

The ultimate efficiency of a cell (adapted from Hisatomi et al. [22]) can be described the half-cell STH for a photoanode:

$$HC - STH = \frac{|I| \times (E_{O_2/H_2O} - E_{RHE})}{P_{sun}} \quad (1)$$

where E_{O_2/H_2O} is the theoretical standard electrode potential for the $O_2 + 4H^+ + 4e^- \rightarrow 2H_2O$ reaction vs. RHE, and E_{RHE} is the pH-dependent potential of the photoanode in the electrolyte, represented as:

$$E_{RHE} = E_{measured} - E_{Ag-AgCl}^0 + \frac{RT \ln 10}{F} pH \quad (2)$$

where $E_{measured}$ and $E_{Ag-AgCl}^0$ are the potential measured relative to an Ag/AgCl reference electrode and the standard potential of the Ag/AgCl reaction, respectively. Since R , T , F are the gas constant, temperature, and, Faraday constant, respectively, cell efficiency is

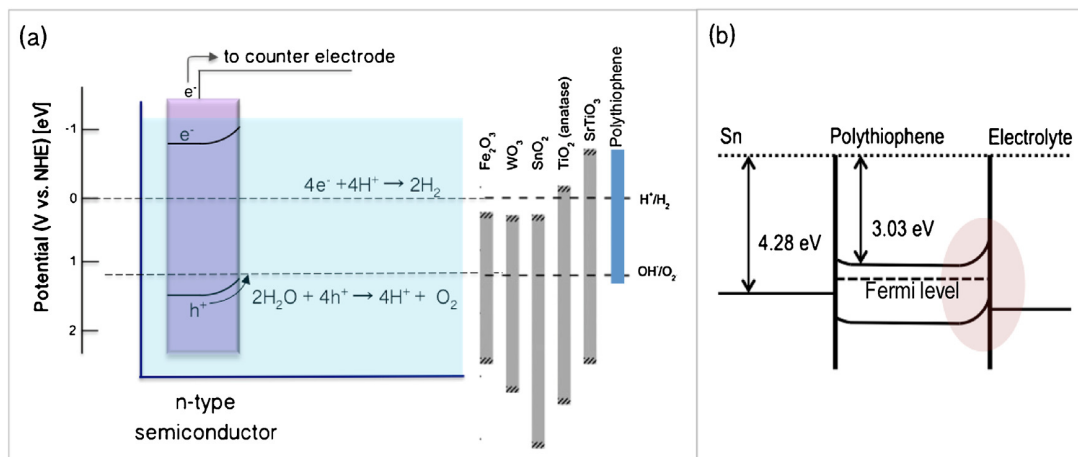


Fig. 2. Representation of a PEC water splitting system: if the conduction band and valence band are above and below the reduction and oxidation potentials, respectively, then charge separation at the surface will allow for water splitting. In an n-type material (shown here), band bending will allow holes to oxidize water, and electrons will reduce resulting hydrogen ions at the counter electrode. By doing so, H₂ gas is produced, which can then take part in the hydrogen economy. For example, fuel cell vehicles will emit H₂O as a by-product, which can then be reused as the aqueous electrolyte in the PEC system.

not only dependent on the measured photocurrent density but also the applied bias potential.

2. Methods

2.1. Device fabrication

A two-step electropolymerization (shown in Fig. 3) process was developed to (1) deposit a seed layer and (2) subsequently polymerize the seed layer as shown in Fig. 4. Alloyed UNS S30100/S30200/S30400 full hardened stainless steel shim (0.002 in. ASTM-A-666, TBI Inc.) is used as the substrate for electropolymerized PT. 0.1 M tetrabutylammonium hexafluorophosphate and 0.1 M thiophene monomer in acetonitrile comprise the electroplating solution. Platinum wire acts as the counter electrode and Ag/AgCl as the reference electrode. Multiple concentrations of thiophene monomer are used to investigate the effects of concentration on the polymerization process. Higher concentrations on the order of 0.1 M and above yield more uniform film thicknesses, with polymerization able to reach the center of a planar substrate. Concentrations on the order of 0.01 M, in contrast, yield no significant electrodeposition. A two-step electropolymerization process

was developed: (1) 10-s seed layer deposition with $V_{\text{bias}} = 2\text{ V vs. Ag/Ag}^+$; and (2) a 60-s film deposition with $V_{\text{bias}} = 1.95\text{ V vs. Ag/Ag}^+$. The conductive electrode is passivated onto a glass substrate using epoxy. The final electropolymerized device is shown in Fig. 5c.

The proposed electropolymerization mechanism is shown in Fig. 3. First, the thiophene monomer combines with an electron becomes a radical cation. The combination of two radical cations form a single dimer of polythiophene, with a by-product of two protons. Since dimers are more easily oxidized than monomers, the dimer then forms its radical form. When a radicalized dimer combines with a radical cation, polymerization begins to occur. It is important to also note that since electron transport occurs much more quickly than monomer diffusion in the electropolymerization electrolyte, there is an assumed high concentration of radicals around the surface of the electrode surface at all times.

A reference P3HT electrode is fabricated by spin coating P3HT dissolved in 1,2 dichlorobenzene or chloroform at varying concentrations from 20 to 30 mg/mL overnight while heated to 70 °C. Spin rate is also varied from 400–800 RPM. Quartz substrates isolate the photocatalytic properties. The electrode is then thermally annealed at 200 °C for 30 min. A top-down view of the spin-coated quartz wafer is shown in Fig. 5b.

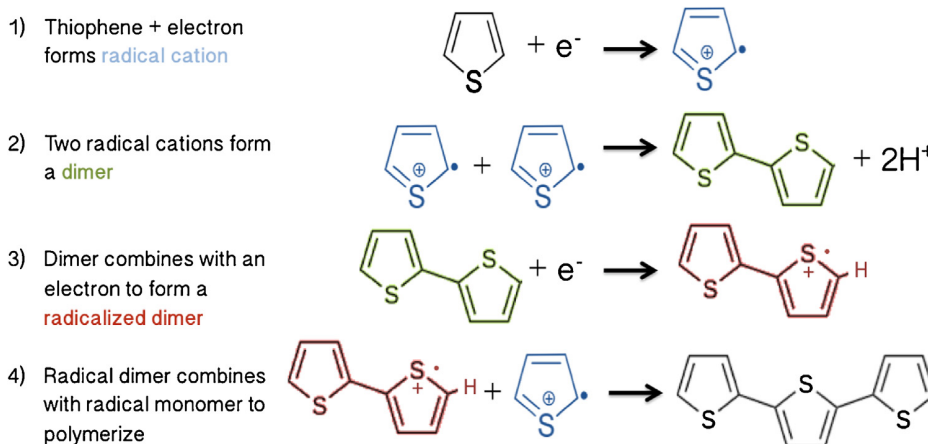


Fig. 3. Under electropolymerization, a constant voltage applied relative to an Ag/AgCl reference electrode and platinum counter electrode, is expected to polymerize thiophene under the following steps: (1) thiophene combines with an electron to form its radical cation, (2) the radical cation combines with another radical cation and forms a dimer + 2 hydrogen ions, (3) a dimer combines with an electron to form its radical form, and (4) the radical dimer combines with a radical monomer to polymerize.

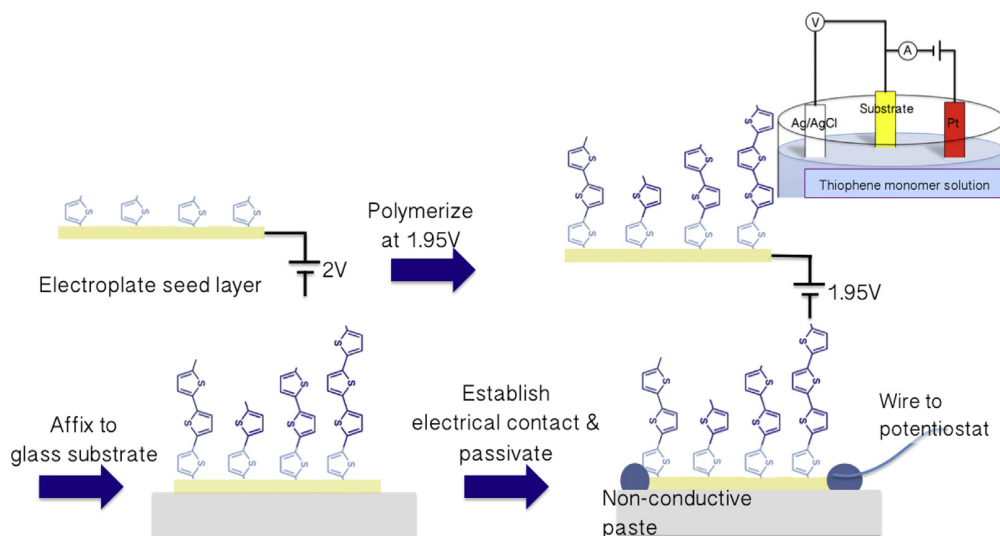


Fig. 4. Fabrication of an electropolymerized thiophene-based electrode. First, a seed layer is deposited at 2V and allowed to polymerize at 1.95V for 60 s on a conductive substrate. After the material is electropolymerized, the substrate is glued and electrically insulated on a glass substrate. Finally, electrical contact is established using conductive tape and insulated wiring. The sample is passivated using epoxy to isolate the active material and to ensure that only the active material is in contact with the aqueous solution.

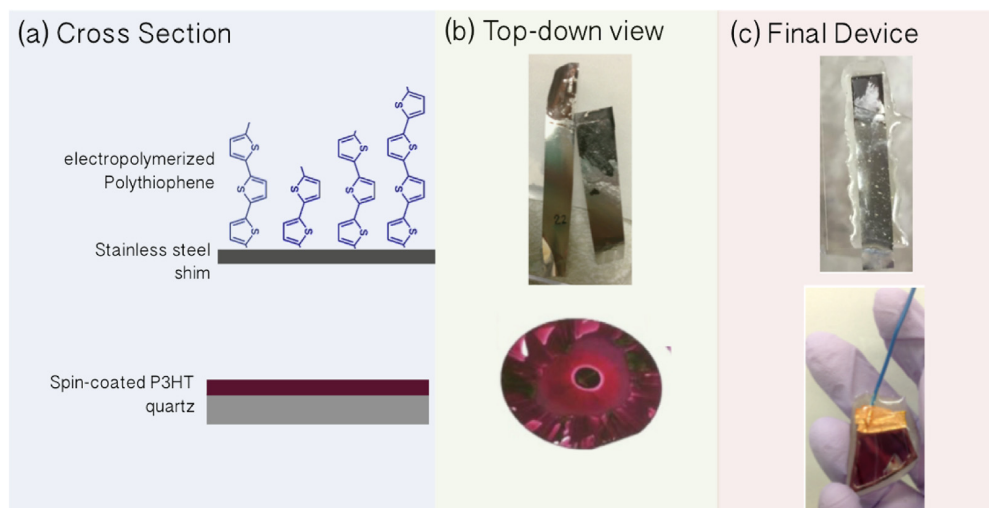


Fig. 5. (a) Cross-sectional view of both electropolymerized PT and spin-coated P3HT on quartz. (b) Top-down view. (c) Final device, with both electrodes fully passivated using epoxy on a glass substrate.

To prepare the final device, the electrode is then affixed onto a glass microscope slide and electrical contact is established using copper tape and insulated tin wire. To electrochemically isolate the P3HT from the copper tape and wire, epoxy was used to passivate the P3HT. The final P3HT device is shown in Fig. 5c. Electrical connection is maintained to the P3HT (front side of the device) rather than its substrate. While quartz was chosen in order to isolate the performance of the polymer material (both electrically and electrochemically), electrodeposition necessitates a conductive substrate for polymerization to take place on the interface of the electrolyte and electrode. That is, electropolymerization on a quartz substrate would be impossible. Thus, stainless steel is chosen for its conductivity and relative electrochemical isolation (does not show water splitting, as compared to substrates such as silicon or copper oxides).

2.2. Electrode testing

The electrode is tested using a three-electrode setup. Electrodes were tested electrochemically using a Ref 600 Gamry Potentiostat. Linear Sweep Voltammetry was conducted in dark and lit conditions under a Xenon lamp (Asahi MAX-303). The electrodes are further isolated from light using an isolation chamber made of Thorlabs Blackout Hardboard. The samples were tested vs. Ag/AgCl from -1 V to 0 V or 0 V to 1 V (depending on majority carrier in the electrode) with a platinum counter electrode in 0.5 M H_2SO_4 electrolyte solution at a scan rate of 50 mV/s.

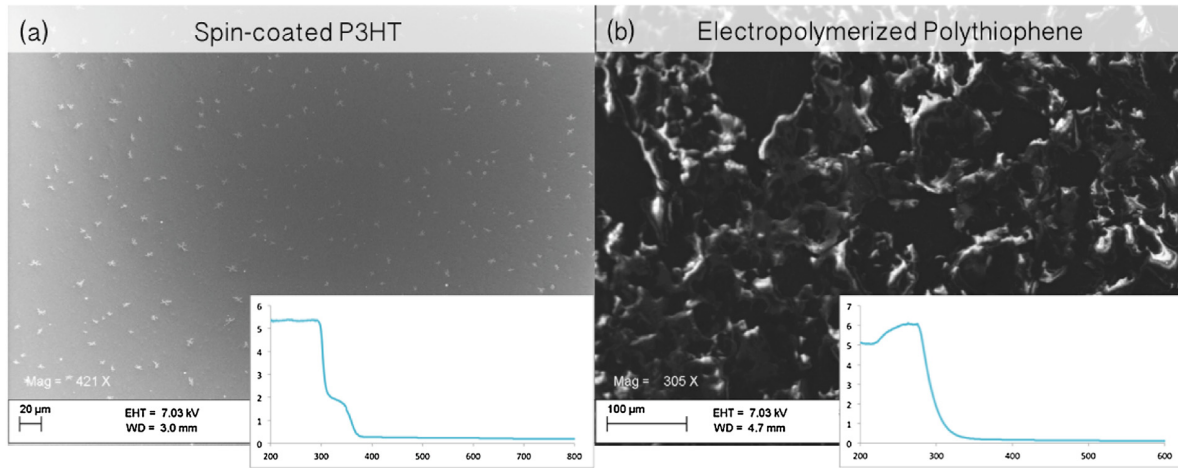


Fig. 6. Fabrication results: SEM images of (a) spin-coated film, with relatively conformal and uniform coverage; (b) electropolymerized polythiophene film, showing increased texturization of the film; (inset) UV/vis absorption spectra for spin-coated P3HT and electropolymerized polythiophene.

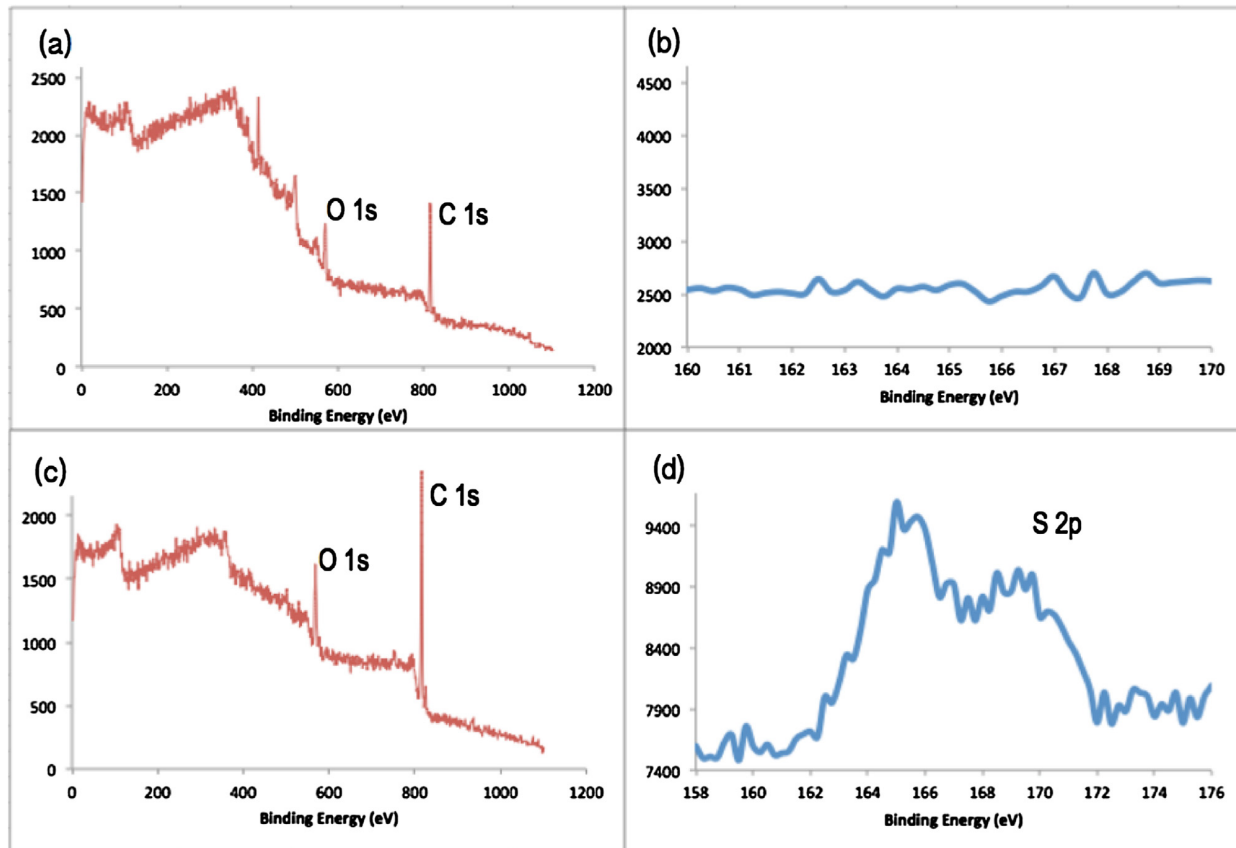


Fig. 7. Characterization by XPS shows (a) full scan of stainless steel shim in a 0.01 M plating solution, (b) scan of sulfur peaks, showing no discernible sulfur, (c) full scan of stainless steel in a 0.1 M plating solution, (d) scan of sulfur. This proves that thiophene detected on the surface was result of electrodeposition.

3. Results and discussion

3.1. Characterization

Scanning electron microscopy (SEM) images of spin-coated P3HT show uniform thickness and conformal coating of the substrate up to hundreds of microns in area (Fig. 7a). A top-down view of the spin-coated P3HT is shown in Fig. 5b. In contrast, electropolymerized PT devices show relatively uneven coverage due to

non-conformal seed layer deposition and polymerization, as shown in Fig. 5b. SEM images of electropolymerized PT also show significant surface texturization, consistent with uneven polymerization across the surface of the stainless steel, as well as pinholes, as seen in the top left corner of Fig. 6b. Furthermore, UV/vis spectra (Fig. 6 inset) demonstrate that the bandgaps of the PT and P3HT are indeed comparable, 3.7 eV and 3.3 eV, respectively. Both materials exhibit high absorption in UV, with optical absorption edge in the UV-vis range. While both materials exhibit a sharp decrease

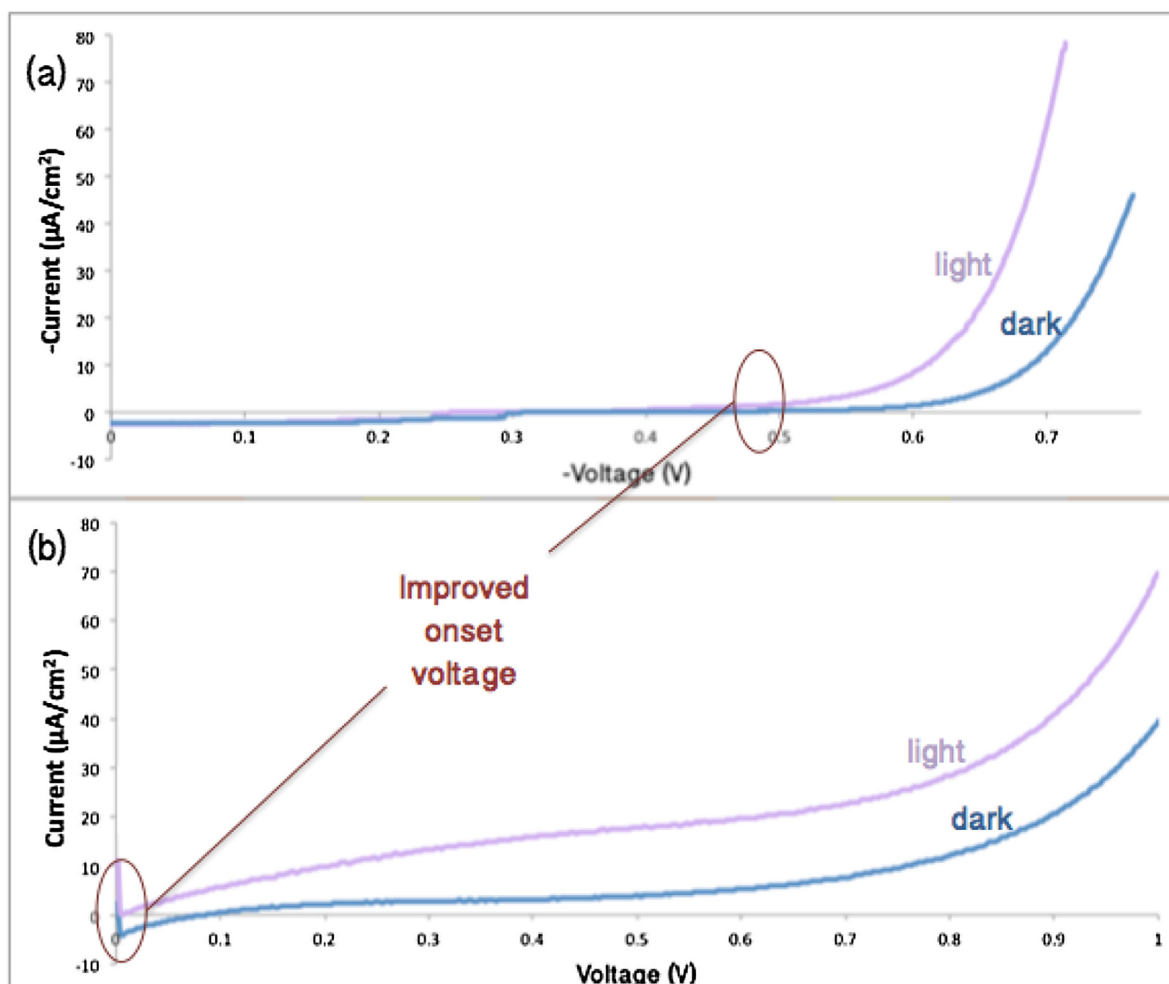


Fig. 8. Photocurrent results for (a) spin-coated, p-type P3HT and (b) electropolymerized, n-type polythiophene, showing significantly improved onset voltage compared to spin-coated P3HT.

in absorption around 300 nm, P3HT exhibits extended absorbance, nearing the visible range.

To confirm electropolymerization, X-Ray Photoelectron Spectroscopy (XPS) was conducted for devices having undergone electropolymerization with a low concentration (0.01 M) thiophene monomer solution and a high concentration (0.1 M) solution. As shown in Fig. 7a and b and discussed in Section 2.1, low concentration solutions failed to yield any significant electroplating of PT, as seen by the lack of noticeable sulfur. In contrast, the XPS for plated thiophene (Fig. 7c) shows significant sulfur peaking with binding energy 162–172 eV (Fig. 7d). This further proves that the presence of PT on the stainless steel shim is not simply residual plating solution, but rather polymerized PT onto the substrate.

3.2. Photocurrent testing

Fig. 8 shows photocurrent measurements of the spin-coated P3HT device and the electropolymerized PT devices. While significant photocurrent is exhibited in the linear sweep voltammetry of both devices, spin-coated devices showed poor onset voltage (~ -0.5 vs. Ag/AgCl). The electropolymerized device shows $V_{on} < 0$ V vs. Ag/AgCl.

The use of electropolymerization allows for increased texturization of the electrode device, by taking advantage of nucleation during the seed layer step. Under SEM, we observe a highly textur-

ized surface on top of the flat conductive substrate. As nucleation tends to occur at point defects on the substrate, the surface of electropolymerized PT exhibits increased surface area. However, as photocurrent is not uniformly larger in the electropolymerized device as compared to that of the spin-coated device, it is unlikely that larger surface area accounts for the entirety of the improved water splitting performance. Moreover, since P3HT shows elevated absorption as compared to that of electropolymerized PT, it is further unlikely that band structure accounts for improved water splitting of the electropolymerized sample.

We posit that our device's superior onset voltage results from improved alignment relative to substrate. Previous literature has shown that the direction of electron flow is dependent on hydrophobicity of elements in the polymer materials, as well as the hydrophobicity of the substrate [14,15]. For example, on a hydrophobic surface, hydrophobic hexyl groups in P3HT are expected to preferentially align to the surface of the substrate, causing electron flow parallel to the surface of the substrate. Similarly, with P3HT on a hydrophilic surface, electron flow is expected to be perpendicular to the substrate surface. Thus, a hydrophilic quartz substrate was chosen for the P3HT: by decreasing the traveling distance of the charge carriers, we expect superior charge separation and water splitting performance.

Since orientation of the polymer relative to the substrate directly affects direction of electron flow, we are able to preclude alignment

of the hydrophobic hexyl groups by electropolymerizing the thiophene monomer. P3HT, even with thermal annealing and substrate choice of quartz, is more difficult to align than PT because grain boundaries around areas of regioregularity in P3HT disrupt electron flow to the substrate (and thus to the counter electrode). That is, P3HT, with hexyl groups, is more likely to be misaligned and hinder electron transport, even when deposited onto a hydrophilic surface to minimize carrier distance. With our electropolymerization method, by precluding alignment, we expect to decrease barriers to carrier flow, allowing for more efficient conversion of solar energy into H_2 – as shown by the excellent onset voltage (0V vs. Ag/AgCl) for the electropolymerized device as compared to the poor onset voltage of the spin-coated device.

Moreover, electroplating has the potential to open pathways toward PT deposition onto porous conductive structures such as activated carbon structures, carbon nanotube forests, and nanowire arrays. By using a highly porous material, one can increase light absorption by using the geometry of the material to harvest reflected light. Porous materials are also able to increase active material while maintaining small minority carrier distances, preserving increased charge separation efficiency.

4. Conclusions

This work presents first demonstrations of electropolymerized organic semiconducting materials (PT) for PEC water splitting. These devices show drastic improvement in V_{on} over conventional spin-coated P3HT devices due to superior charge separation and texturization using the electropolymerization method. As compared to spin-coated P3HT, with $V_{on} \sim -5.5$ V vs. Ag/AgCl, EP-PEC devices achieve $V_{on} = 0$ V vs. Ag/AgCl. As such, electroplating has the potential to open pathways toward more robust deposition of organic semiconductor PEC materials onto porous conductive substrates.

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Biographies



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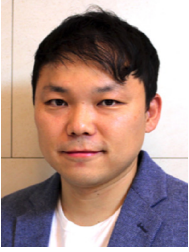
Qiaohao Liang is an undergraduate student researcher working under Professor Liwei Lin in the Mechanical Engineering Department at the University of California, Berkeley. He is currently pursuing his B.S. degree in Material Science and Engineering at University of California, Berkeley. Integrating computational tools into the domain of material science, he has a wide interest in designing and synthesizing new materials. He also enjoys blowing the whistle and making calls as a basketball referee.



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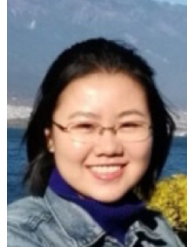
Xining Zang is a postdoc researcher in Prof Liwei Lin group, mechanical engineering of UC Berkeley. She has been a BSAC graduate researcher since 2012, and she got her PhD degree in 2017 August. Her research interest spans from 2D materials synthesis and assembly to nanostructured device. Her current research focus on wafer scale manufacturing and integration of 2D materials including transition metal chalcogenide and transition metal carbides for electronics and energy related applications (catalyst, battery and supercapacitors). She also develops a consecutive research interest in on-paper fabrication and functional materials integration for origami structured disposable paper electronics.



Hyun Sung Park received his M.S. degree in Mechanical and Aerospace Engineering from Seoul National University, South Korea in 2006 and received his Ph.D. degree in Mechanical engineering from UC Berkeley, the USA in 2017. He performed research on the metal oxide and its applications for the solid state electronics. He is currently a senior engineer of Samsung Display Company and has extensive experience in multi-physics simulation knowledge. His research interests include device physics and sensor embedded display devices with a focus on emerging technologies for flexible and stretchable electronics.



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Jianan Lu is a fourth-year undergraduate majoring in computer science at University of California, Berkeley. She has a wide range of academic interests, including computer security, privacy and innovative energy storage. In her free time, she likes to explore the nature and travel.



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