

# Enhanced efficiency in dye-sensitized solar cells based on mesoporous titanium phosphate photoanode



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## ABSTRACT

A new bilayer-nanostructured photoanode with mesoporous titanium phosphate (MTP1) as overlayer and titanium dioxide (TiO<sub>2</sub>) as underlayer has been fabricated. Mesoporous titanium phosphate material with high surface area (442 m<sup>2</sup> g<sup>-1</sup>) has been synthesized hydrothermally by using a new tailor made gemini surfactant under acidic pH conditions. HR TEM and N<sub>2</sub> sorption analysis revealed the presence of disordered mesopores of dimension ca. 4.50 nm in MTP1 material. Open-circuit voltage decay (OCVD) spectroscopy is used to investigate the kinetic process of electron transport and recombination within TiO<sub>2</sub>/TiO<sub>2</sub>, MTP1/TiO<sub>2</sub>, or MTP1/MTP1 based DSSCs, revealing a faster electron transport rate and a lower recombination rate in the MTP1/TiO<sub>2</sub> photoanode. The energy conversion efficiency of dye-sensitized solar cells (DSSCs) based on such a MTP1/TiO<sub>2</sub> photoelectrode shows a significant enhancement (6.0%) compared to TiO<sub>2</sub>/TiO<sub>2</sub> photoelectrode (5.5%) with similar thickness (~7 μm), suggesting the favorable electron transport rate in former.

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## 1. Introduction

With the rapidly diminishing fossil fuel reserves the scientists, entrepreneurs and government leaders are keen to find a cheap, clean and sustainable alternative energy resource [1]. In this context dye-sensitized solar cells (DSSCs) have attracted intense scientific attention due to their advantages over conventional Si-based solar cell devices, in terms of flexibility in cell design, reduced cost, light weight, and the low toxicity of the manufacturing process [2]. DSSCs are typically made of two transparent conducting glasses, one of which is a photoanode and the other is a counter electrode, with an iodide electrolyte placed between the electrodes [3–11]. TiO<sub>2</sub> has traditionally been employed the primary material for the photoanode since the initial fabrication of the DSSC proposed by Graetzel et al. [7]. At the photoanode, light absorption by dye molecules results in the injection of the electrons into the conduction band of the porous TiO<sub>2</sub>. The photoanode, which is one of the main components in DSSCs, is usually fabricated using semiconductor nanomaterials such as TiO<sub>2</sub>, due to their large surface area to volume ratios [9,10]. However, the

conversion efficiency of TiO<sub>2</sub> nanoparticles-based DSSCs is limited due to the slow transportation of electrons through the randomly arranged particles.

Titanium/zirconium phosphates are expected to exhibit faster electron transport due to framework charge [12–16]. They are cheap, abundant in nature and easy to prepare via fast crystallization and anisotropic growth making them excellent candidates for DSSCs with photoanodes having nanostructures like nanorods, nanowires, nanotubes, nanotetrapods or nanosheets. Although, there have been many reports on layered [16] and open-framework titanium phosphates, there are only few reports on porous titanium phosphates with high surface areas [17–19]. Designing of materials with both porosity and controlled layered structures [20,21] of various compositions are very important for exploring their application in energy research. Specially, porosity and nanostructure of the photoanodes/counter electrodes play crucial role in optoelectronic applications [22–25]. Thus, use of novel mesoporous semiconductor materials [26–28] for the construction of photoanodes in a DSSC is very challenging.

In this context controlling the nanoscale morphology of the TiO<sub>2</sub> photoanode to minimize electron recombination is crucial to improve the efficiency of DSSCs [29,30]. Chen et al. reported that layer-by-layer (LbL) assembly via thin film deposition method

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offers several advantages in designing an efficient photoanode [10]. It is a simple and cost-effective process with strong potential for scalability. Equally important that it allows for a high degree of nanoscale control over the thickness, morphology, and chemical composition of the resulting thin films [16]. Thus, considerable efforts are underway for harnessing multiple interfaces to enhance the performance of a DSSC [31].

However, the solar cell performances based on these structures reported till date is still far below those of nanoparticle-based TiO<sub>2</sub> photoanodes [32] due to low specific surface area and/or other limiting factors. In general, high specific surface area and fast electron transports are indispensable to a high-performance photoanode, but these factors are often incompatible with one another. To accommodate all of these favorable characteristics, we propose a double-layered film structure with two different materials for DSSC photoanodes. Further, to study the dynamics behind such high open circuit voltage in MTP1 electrode, photocurrent decay measurements are performed.

## 2. Experimental

### 2.1. Materials

1,5-Dibromopentane, *N,N*-dimethyltetradecyl amine and titanium isopropoxide were purchased from Sigma–Aldrich, ortho-phosphoric acid (GR, 85% in water) was obtained from Merck, and EtOH, HCl and ammonia (25% aqueous) were purchased from Avra Chemicals, India. All the chemical were used without any further purification.

### 2.2. Preparation of gemini surfactant S<sub>14-5-14</sub>

The cationic gemini surfactant pentamethylene-1,5-bis(dimethyltetradecylammonium bromide) (S<sub>14-5-14</sub>, Scheme 1) was synthesized by refluxing 1,5-dibromopentane with *N,N*-dimethyltetradecyl amine in dry ethanol for 48 h [33,34]. Then the solvent was evaporated and the product was recrystallized several times from ethanol/acetone mixture. Its structure was confirmed by mass spectroscopy and <sup>1</sup>H NMR spectroscopy. The purity of the gemini surfactant was verified by elemental analysis and the absence of minima in surface tension ( $\gamma$ ) vs. log [surfactant] plot [35–37].

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz):  $\delta$  (ppm): 0.75–0.92 (t, terminal CH<sub>3</sub>, 6H), 1.17–1.33 (br, (CH<sub>2</sub>)<sub>11</sub>, 44H), 1.54–1.75 (m, CH<sub>2</sub>, –CH<sub>2</sub>–CH<sub>2</sub>–(CH<sub>2</sub>)<sub>11</sub>–, 4H), 1.96–2.08 (br,  $\gamma$ -CH<sub>2</sub>– in the spacer,

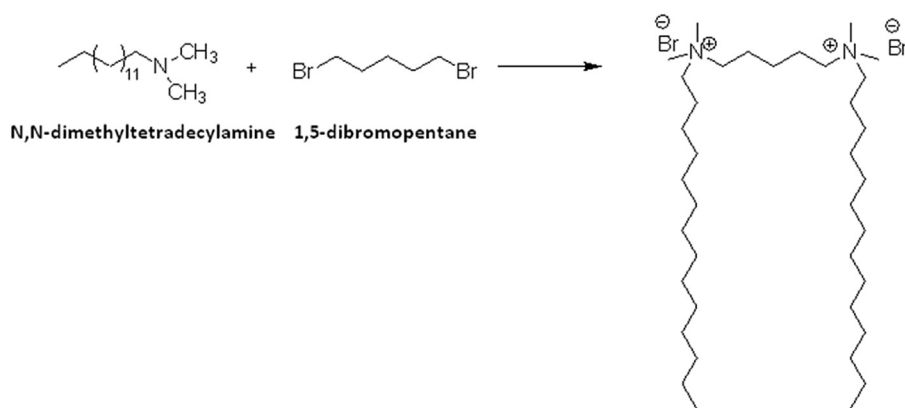
2H), 3.28 (s,  $\beta$ -CH<sub>2</sub>, in the spacer, 4H), 3.29–3.38 (t, –N(CH<sub>3</sub>)<sub>2</sub>–CH<sub>2</sub>–CH<sub>2</sub>–, 4H), 3.63–3.67 (t,  $\alpha$ -CH<sub>2</sub>, in the spacer, 4H), 3.92 (s, –N(CH<sub>3</sub>)<sub>2</sub>–CH<sub>2</sub>, 12H). C, H, N analysis, calcd. for C<sub>37</sub>H<sub>80</sub>N<sub>2</sub>Br<sub>2</sub>: C 62.34, H 11.32, N 3.93. Found C 62.44, H 10.96, N 3.88. The mass to-charge ratios [38] for the gemini surfactant S<sub>14-5-14</sub> [i.e. (M<sup>2+</sup>/2) + H] is 356.1946, which is found to be very close to the calculated value of 356.2344 (Supporting information, Fig. S1).

### 2.3. Synthesis of mesoporous titanium phosphate (MTP1)

498 mg (0.7 mmol) of cationic gemini surfactant S<sub>14-5-14</sub> was dissolved in 5 ml of water and then 0.23 ml of ortho-phosphoric acid (85%) was added to it and homogenized with continuous stirring for 15 min. Then 568.52 mg (2 mmol) of titanium isopropoxide taken in an equal amount (w/w) of isopropyl alcohol was added dropwise for 10 min. After stirring of this reaction mixture for 1 h at room temperature the pH of the medium was adjusted to ca. 5.0 using liquid ammonia and then the resultant slurry was transferred to an autoclave and heated at 348 K for 1 day under autogenous pressure [11]. After the hydrothermal synthesis the product was filtered and washed thoroughly with distilled water and dried in an oven at 348 K. To remove the surfactant from the as-synthesized material, the mesoporous titanium phosphate sample was extracted with EtOH/HCl mixture for 4 h at room temperature.

### 2.4. Fabrication of DSSC

A transparent film made of a TiO<sub>2</sub> paste with about 18-nm-sized particles (18-NRT, Dyesol) was deposited by the doctor-blade on top of conductive fluorine-tin-oxide (FTO) glass substrates (8  $\Omega$  cm<sup>-2</sup>, Pilkington). The MTP1 paste was prepared from synthesized MTP1 nanoparticles, ethylcellulose binder (45 cP, Kanto Chemical), and  $\alpha$ -terpineol solvent (Sigma–Aldrich) in a weight ratio of 20:10:70. The MTP1 paste was then coated onto FTO substrate by the doctor-blade technique. After pre-cleaning the FTO glass, the doctor blade of the TiO<sub>2</sub> paste was conducted double layer (TiO<sub>2</sub>/TiO<sub>2</sub> electrode), MTP1 paste was coated on TiO<sub>2</sub> paste coated film (MTP1/TiO<sub>2</sub>) and MTP1 paste double coated film (MTP1/MTP1 electrode). Between each print, the FTO glass was heat treated at 450 °C for 30 min. The printed FTO glass was used as photo-electrode in DSSC after immersion in a 5  $\times$  10<sup>-4</sup> mol/L ethanol solution of Ru(dcbpy)<sub>2</sub>(NCS)<sub>2</sub> (N719, Solaronix) for 6 h and subsequent aging in a dark room for 24 h. Pt-coated FTO glass, which was heat-treated at 450 °C for 30 min after coating, was used as a counter electrode. The electrolyte composition was 0.05(M) I<sub>2</sub> (Aldrich, 99.99%), 0.5(M) Lil



Scheme 1. Synthesis of gemini surfactant S<sub>14-5-14</sub>.

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