Promoting Photoelectrochemical Water Oxidation on Ti-doped Fe$_2$O$_3$ Nanowires Photoanode by O$_2$ Plasma Treatment

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1. Supporting Figures

The conditions of O$_2$ plasma treatment were optimized first. The linear scan voltammetry (LSV) curves of pristine Ti-Fe$_2$O$_3$ and OPT-Ti-Fe$_2$O$_3$ photoanodes are performed in 1 M NaOH (pH=13.6) aqueous solution at a scan rate of 10 mV s$^{-1}$, and the results were presented in Figure S1. From Figure S1a, it can be seen that pristine Ti-Fe$_2$O$_3$ photoanode yielded a photocurrent density of 1.10 mA·cm$^{-2}$ at 1.23 V vs. RHE. OPT-Ti-Fe$_2$O$_3$ photoanodes treated under 100 W for different time of 90 s, 150 s, and 210 s all exhibited almost the same photocurrent of 1.65 mA·cm$^{-2}$, which is 1.5 times higher than pristine Ti-Fe$_2$O$_3$. The onset potential of OPT-Ti-Fe$_2$O$_3$ photoanodes had a slightly negative shift of ~20 mV compared with pristine Ti-Fe$_2$O$_3$. The similar LSV curves of the samples indicate that the samples did not change significantly when the treated time increased from 90 to 210 s.

When the power of O$_2$ plasma was increased to 200 W, the photocurrent densities of OPT-200W-50s, OPT-200W-70s, and OPT-200W-90s photoanodes were further increased to 1.87, 2.14, and 1.69 mA·cm$^{-2}$ at 1.23 V vs. RHE, respectively, as depicted in Figure S1b. OPT-200W-70s photoanode has the highest photocurrent density, which was 1.95 times that of pristine Ti-Fe$_2$O$_3$. The onset potential of OPT-200W-70s photoanode exhibited an approximate 50 mV cathodic shift compared with pristine Ti-Fe$_2$O$_3$, which lowered the applied potential to drive the PEC water oxidation reaction. It was also found that the photocurrent of OPT-200W-70s here showed superiority for water oxidation than many
reported Ti-Fe₂O₃ nanowires as depicted in Table S1. These all suggested that O₂ plasma treatment is an effective method for promoting the PEC water oxidation kinetics of Ti-Fe₂O₃ photoanodes.

![Image](https://example.com/image.png)

**Figure S2.** I-t curves of pristine Ti-Fe₂O₃ and OPT-200W-70s photoanodes.

**2. Supporting Table**

Table S1. The structure, fabrication method, and photocurrent density of some reported works on Ti-doped a-Fe₂O₃ nanowires photoanodes.

| Structure (Ti-Fe₂O₃) | Fabrication method | Photocurrent density | Ref. |
|----------------------|--------------------|----------------------|------|
| Ti-Fe₂O₃ nanorod     | Hydrothermal       | 1.5 mA cm⁻²          | 5    |
|                      | / RF magnetron sputtering | (1.23 V vs. RHE)     |      |
|                      |                    | (AM 1.5G/100 mW cm⁻²)|      |
| Ti-Fe₂O₃ nanorod/Co-MOF cocatalyst | Hydrothermal/dip-coating | 1.5 mA cm⁻²  | 19 |
|                      |                    | /2.24 mA cm⁻²        |      |
|                      |                    | (1.23 V vs. RHE)     |      |
|                      |                    | (AM 1.5G/100 mW cm⁻²)|      |
| Ti-Fe₂O₃ nanorod/Co-Pi | Hydrothermal combined wet-chemical impregnation/photo-assisted electrodeposition | 0.8 mA cm⁻² | 20 |
|                      |                    | (1.23 V vs. RHE)     |      |
|                      |                    | (500 W Xenon lamp/100 mW cm⁻²)|      |
| Ti-Fe₂O₃ nanorod/Cr-doped SrTiO₃ | Hydrothermal/Hydrothermal | 1.09 mA cm⁻² | 21 |
|                      |                    | (1.23 V vs. RHE)     |      |
|                      |                    | (500 W Xenon lamp)   |      |
| Ti-Fe₂O₃ nanorod/Pt | Hydrothermal/Immersion method | 1.56 mA cm⁻² | 22 |
|                      |                    | (1.23 V vs. RHE)     |      |
|                      |                    | (AM 1.5G/100 mW cm⁻²)|      |
| Ti-Fe₂O₃ nanowires array | Hydrothermal/O₂ plasma treatment | 2.14 mA cm⁻² | (this work) |
|                      |                    | (1.23 V vs. RHE)     |      |
|                      |                    | (AM 1.5G/100 mW cm⁻²)|      |

**3. Characterization and Photoelectrochemical experiments**

The Ti-Fe₂O₃ thin films were characterized by X-ray diffraction with a Rigaku D/max-2500 using Cu Ka radiation (λ = 1.5406 Å) and Raman spectra by J-Y UV-visible micro-Raman spectrometer with excitation at 488 nm from an Ar⁺ laser. The optical properties were collected by the Perkin Elmer UV Win Lab spectrometer with an integrating sphere. X-ray photoelectron spectroscopy (XPS) measurements were collected with a PHI 5000
VersaProbe (ULVAC-PHI) using an Al Kα X-ray source (1486.6 eV). The electron analyzer was operated at pass energies of 117.4 eV for the wide scans and 23.5 eV for the narrow scans.

All the photoelectrochemical (PEC) experiments were carried out in a three-electrode configuration via a Princeton 2273 electrochemical workstation. Pristine and OPT-TiFe2O3 films were used as working electrode, Pt black was used as the counter electrode. Ag/AgCl (3.5 M KCl) was used as the reference electrode. All the surface area of the photoanodes was approximately 1.22 cm². A Newport simulated solar irradiation (LCS-100, 100 mWcm⁻²) was used as the light source. The Mott-Schottky curves were measured in the dark at a constant frequency of 1 kHz with a step of 50 mV. The electrochemical impedance spectroscopy (EIS) was measured under light conditions with a frequency ranging from 100 kHz to 0.01 Hz. The AC amplitude was set as 10 mV. The PEC measurements were conducted in 1 M NaOH aqueous solution, and 1 M NaOH with 0.5 M H₂O₂ was used as the electrolyte for the analysis of charge transfer efficiency at the interface of electrode/electrolyte.