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Construction of Heterostructured Fe₂O₃-TiO₂ Microdumbbells for Photoelectrochemical Water Oxidation

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Abstract

Designing advanced structures for heterojunction photocatalysts is an effective approach to enhance their performance toward solar energy conversion. Here we develop a facile synthetic strategy for the fabrication of Fe₂O₃-TiO₂ microdumbbells. With the assistance of preferentially adsorbed hexadecylamine molecules, amorphous TiO₂ nanospheres with tunable size are grown at the two ends of Fe-based metal-organic compound microrods. Subsequent annealing of the obtained hybrid leads to the formation of a novel heterostructured Fe₂O₃-TiO₂ microdumbbell photocatalyst. Due to the formed heterojunction and unique structure, Fe₂O₃-TiO₂ microdumbbells with optimized composition and morphology show enhanced performance for photoelectrochemical water oxidation, compared to monophasic Fe₂O₃ and TiO₂ materials as well as physical mixtures thereof.

Keywords: metal-organic compounds; heterostructures; microdumbbells; Fe₂O₃, TiO₂.

Developing clean and renewable energy sources is of great importance for modern society due to the limited reserves of fossil fuels and the serious environmental issues caused by excess carbon emissions.^[1,2] Among renewable energy sources, solar energy is one of the most promising candidates because it is universal, benign, inexhaustible, and has high potential capacity.^[3] However, the decentralized and intermittent nature of insolation is still a great barrier to the practical application of solar energy conversion.^[4] To solve these problems, direct conversion of solar energy into chemical fuels, mimicking photosynthesis in nature, is an attractive approach.^[5,6] Photoelectrochemical (PEC) water splitting is a highly promising strategy to generate hydrogen energy, which can be stored, transported and widely utilized in clean and sustainable applications.^[7,8] Therefore, it is essential to design high-performance, cost-effective and robust photocatalysts for efficient PEC water splitting systems.^[9]

Semiconductor materials with excellent chemical, physical and electrical properties have been extensively investigated as photocatalysts for solar water splitting.^[10] However, identifying a single semiconductor that meets all the requirements for high activity, including a suitable band gap structure, low charge resistance, fast surface reaction kinetics, and good stability, has proven challenging.^[11] The synthesis of heterojunction photocatalysts by combining semiconductor materials with different band gap structures has become an important approach to enhance the efficiency of PEC cells.^[12,13] In a heterojunction photocatalyst, internal electric field can be built between different components, which would promote the spatial separation of photogenerated electron-hole pairs.^[14,15] Moreover, broadband light absorption can be realized if the heterojunction photocatalyst contains semiconductors responsive to different portions of the solar spectrum.^[16,17] The fabrication of highly active heterojunction photocatalysts requires the careful selection of appropriate building blocks.^[18]

Since the seminal work by Fujishima and Honda, semiconducting TiO_2 has been widely studied as an important photocatalyst for PEC water splitting, which shows relatively high activity and stability under ultraviolet-light (UV-light).^[19,20] Fe_2O_3 is also an attractive photocatalyst with a band gap of 1.9-2.2 eV and good performance under visible-light (Vis-light).^[21-24] These two earth-abundant, non-toxic and robust semiconductor materials are good candidates for the construction of heterojunction photocatalysts with high charge separation efficiency and good light absorption ability,^[25,26] leading to enhanced performance for PEC water oxidation.^[27,28] Therefore, tuning the composition and structure of Fe_2O_3 - TiO_2 heterojunction photocatalysts is important for optimizing their performance toward photocatalytic reactions.^[29,30]

Herein, we have developed a facile synthetic strategy to fabricate Fe_2O_3 - TiO_2 microdumbbells for PEC water oxidation (**Figure 1a**). Specifically, Fe-based metal-organic compound (FeMOC) microrods are first synthesized by a hydrothermal approach with polyvinylpyrrolidone (PVP) as the surfactant (see Supporting Information for experimental details). Then, amorphous TiO_2 (Am TiO_2) nanospheres are grown at the two ends of the FeMOC microrods through a solution reaction to generate FeMOC-Am TiO_2 microdumbbells. Annealing of the as-prepared composite leads to the formation of Fe_2O_3 - TiO_2 microdumbbells, which exhibit enhanced performance for PEC water oxidation compared with Fe_2O_3 microrods, TiO_2 nanospheres and physical mixtures of the two species. Compared with traditional approaches to fabricate photocatalysts, the developed synthetic strategy opens up new avenues toward the design of photoelectrode materials with well-controlled configurations and advanced catalytic properties.

Field-emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) images show that the obtained FeMOC microrods are uniform with a high aspect ratio (Figure 1b-d). The sharp diffraction peaks at 2θ of 9.3° , 10.7° , 14.2° and 16.1° in the X-ray diffraction (XRD)

pattern indicate the FeMOC microrods have good crystallinity (Supporting Information, Figure S1a), although the exact crystal structure remains unknown. As revealed by energy-dispersive X-ray spectroscopy (EDXS), the FeMOC microrods consist of Fe, C and O elements (Supporting Information, Figure S1b). Thermogravimetric (TG) analysis data show that nearly 60% of the initial weight of FeMOC microrods is lost at 300 °C (Supporting Information, Figure S1c), which indicates that the compound is constructed by Fe nodes and organic ligands.^[31]

Subsequently, AmTiO₂ nanospheres with the size of about 450 nm are grown at the two ends of the FeMOC microrods through a solution reaction with the assistance of hexadecylamine (HDA). The obtained sample is denoted as FeMOC-AmTiO₂-40, where the suffix refers to the amount of HDA (in mg) used during synthesis; this applies to all sample naming. FeMOC-AmTiO₂-40 possesses a well-defined structure of microdumbbells (Figure 1e,f). TEM imaging of a FeMOC-AmTiO₂-40 microdumbbell reveals the close connection between the FeMOC microrod and the AmTiO₂ nanosphere (Figure 1g). The spatially-selective growth of AmTiO₂ nanospheres is further confirmed by high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) and elemental mapping images, which show that a Ti-enriched nanosphere is preferentially formed at one end of the FeMOC microrod (Supporting Information, Figure S2). The XRD pattern of the FeMOC-AmTiO₂-40 microdumbbells shows similar characteristic peaks to the FeMOC microrods, indicating the amorphous nature of the TiO₂ nanospheres (Supporting Information, Figure S3a). The Fe/Ti molar ratio of the FeMOC-AmTiO₂-40 microdumbbells is about 4 (Supporting Information, Figure S3b).

The as-prepared FeMOC-AmTiO₂-40 is converted into Fe₂O₃-TiO₂-40 by annealing in air, during which the microdumbbell structure is preserved (**Figure 2a-c**). TEM imaging of a Fe₂O₃-TiO₂-40 microdumbbell reveals the mesoporous property of the Fe₂O₃ microrod as a result of the removal of organic ligands in the FeMOC microrods during the heat treatment (Figure 2d). High-resolution TEM

(HRTEM) imaging (Figure 2e) demonstrates that the heterostructure is constructed by anatase TiO_2 nanocrystals with lattice spacing of 0.24 nm corresponding to the (004) planes and Fe_2O_3 nanocrystals with lattice spacing of 0.37 nm for the (012) planes, which is consistent with the results of XRD and EDXS characterizations (Supporting Information, Figure S4).^[32,33] As revealed by X-ray photoelectron spectroscopy (XPS), Fe(III) and Ti(IV) are formed after the annealing process without the generation of other oxidation state or impure species (Supporting Information, Figure S5). Moreover, formation of the Fe_2O_3 - TiO_2 microdumbbells is further confirmed by the HAADF-STEM and elemental mapping images, showing that the TiO_2 nanosphere wraps the end of the Fe_2O_3 microrod with the formation of a large cylindrical interface (Figure 2f). The tight contact between Fe_2O_3 and TiO_2 might be beneficial for the migration of charge carriers at the heterojunction during photocatalytic reactions.

Since HDA plays a very important role in the growth of Am TiO_2 nanospheres,^[34] the formation mechanism of the microdumbbells is investigated via experiments with varying amounts of HDA surfactant. Without the addition of HDA in the reaction solution, Am TiO_2 could not form on the FeMOC microrods (**Figure 3a** and Supporting Information, Figure S6a). When 10 mg of HDA is used, small Am TiO_2 particles are observed at the ends of the FeMOC microrods (Figure 3b and Supporting Information, Figure S6b). The Am TiO_2 particles become larger and more spherical with the increase of HDA to 20 mg, the size of which further enlarges by using 40 mg of HDA (Figure 3c,d and Supporting Information, Figure S6c). However, morphology of the Am TiO_2 particles turns to be irregular if the amount of HDA is increased to 80 mg and 120 mg (Figure 3e,f and Supporting Information, Figure S6d,e).

Based on these observations, the formation mechanism of the FeMOC-Am TiO_2 microdumbbells is proposed (**Figure 4**). During the synthesis of FeMOC microrods, PVP molecules tend to cover the surfaces. Thus, HDA molecules may be blocked by the PVP and preferentially adsorb on the ends of

the FeMOC microrods. Since HDA can interact with AmTiO₂ formed by the hydrolysis of titanium isopropoxide through hydrogen-bonding, AmTiO₂ nanospheres can be grown selectively at the ends leading to the formation of FeMOC-AmTiO₂ microdumbbells.^[34] When the amount of HDA is insufficient (e.g., 10 mg in the reaction system), only small AmTiO₂ particles can be formed as a result of the limited surfactants for bonding at the ends of the FeMOC microrods. However, excessive free HDA molecules (when 80 mg of HDA is added) in the reaction solution may lead to overgrowth of the AmTiO₂ phase and generation of irregularly shaped large particles. To further verify the hypothesized mechanism, FeMOC microrods synthesized without PVP are used for the growth of AmTiO₂ (Supporting Information, Figure S7a,b). With 40 mg of HDA, AmTiO₂ particles randomly grow on the surface of the bare FeMOC microrods (Supporting Information, Figure S7c,d), demonstrating the critical blocking effect of PVP and the necessity of the preferential adsorption of HDA for the fabrication of FeMOC-AmTiO₂ microdumbbells. Therefore, the blocking effect of PVP, selective adsorption of HDA at the ends, and the strong interaction between HDA and AmTiO₂ are essential for the formation of FeMOC-AmTiO₂ microdumbbells.

In order to reveal the advantages of the Fe₂O₃-TiO₂ heterojunction photocatalyst for PEC water oxidation, Fe₂O₃ microrods, TiO₂ nanospheres, and their physical mixture (denoted as Fe₂O₃-TiO₂-Mix, with the same composition as Fe₂O₃-TiO₂-40) are prepared as control samples (Supporting Information, Figure S8 and S9). Besides, annealing of the FeMOC-AmTiO₂ samples fabricated with different amounts of HDA results in the formation of Fe₂O₃-TiO₂ heterojunction photocatalysts with different compositions (Supporting Information, Figure S10). The PEC performance of the samples is evaluated using a three-electrode cell containing 1.0 M NaOH electrolyte. The low dark current densities demonstrate that the photocatalysts are almost inactive without irradiation (Supporting Information, Figure S11). Photocurrent density versus bias (*J-V*) curves are collected under simulated air mass 1.5

global (AM 1.5G) irradiation with an intensity of 100 mW cm^{-2} . Compared with bare Fe_2O_3 microrods and TiO_2 nanospheres, $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-Mix}$ shows enhanced photocurrent density, indicating that the heterojunction between Fe_2O_3 and TiO_2 can promote water oxidation activity (**Figure 5a**). However, the photocurrent density of $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ is much higher than that of $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-Mix}$, which reveals the superiority of the well-defined structure with tight heterojunction for PEC water oxidation. Incident photon-to-current conversion efficiencies (IPCEs) are measured under monochromic irradiation (Figure 5b). $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ microdumbbells exhibit the highest IPCEs under both UV-light and Vis-light. This result indicates that the heterojunction between the UV-light-responsive TiO_2 and Vis-light-responsive Fe_2O_3 can promote the separation of charge carriers (Supporting Information, Figure S12 and S13) and enhance the light absorption ability of the photocatalyst (Supporting Information, Figure S14). The composition and structure of the heterojunction photocatalyst are important factors to affect its performance. The $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-20}$ sample with a higher content of TiO_2 shows better performance than $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-10}$, while the highest photocurrent density is achieved by $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ microdumbbells with the optimized composition and structure (Figure 5c). Further increasing the fraction of TiO_2 would reduce the activity. This phenomenon might be caused by the excess recombination centers induced by the irregular structures of $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-80}$ and $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-120}$, which is proved by their high photoluminescence (PL) signals (Supporting Information, Figure S13). IPCEs of $\text{Fe}_2\text{O}_3\text{-TiO}_2$ heterojunction photocatalysts show a trend similar to the J - V curves (Figure 5d). $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ microdumbbells show good stability for PEC water oxidation with no noticeable deactivation over 4 days (Supporting Information, Figure S15). The PEC performance of the $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ microdumbbells compares favorably to those of reported representative $\text{Fe}_2\text{O}_3\text{-TiO}_2$ heterojunction photocatalysts for PEC water oxidation.^[35-37] However, further improvement is still needed for the

present system, since the charge transfer between the photocatalysts and the substrate may not as good as thin-film photoelectrodes (Supporting Information, Table S1).^[27,28,38]

In summary, novel Fe₂O₃-TiO₂ microdumbbells have been fabricated through a facile synthetic approach. Fe-based metal-organic compound (FeMOC) microrods are first synthesized through a hydrothermal reaction, at the ends of which amorphous TiO₂ (AmTiO₂) are grown to form FeMOC-AmTiO₂ microdumbbells. A subsequent annealing process results in the formation of Fe₂O₃-TiO₂ microdumbbells, which show enhanced performance and stability for photoelectrochemical water oxidation. By designing control experiments using varied amounts of surfactants, formation mechanism of the heterostructured microdumbbells is also proposed. This mechanism may guide the development of synthetic strategies for the fabrication of novel hybrid materials and inspire further capability on constructing complex heterojunction photocatalysts for efficient solar energy utilization.

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Figures and Captions

Figure 1. (a) Schematic illustration of the Fe₂O₃-TiO₂ microdumbbell formation process. (i) Selective solution growth of AmTiO₂ spheres at the two ends of a FeMOC microrod to form FeMOC-AmTiO₂ microdumbbell. (ii) Annealing of the obtained FeMOC-AmTiO₂ microdumbbell to generate Fe₂O₃-TiO₂ microdumbbell. (b,c) FESEM and (d) TEM images of FeMOC microrods. (e,f) FESEM and (g) TEM images of FeMOC-AmTiO₂-40 microdumbbells.

Figure 2. (a-c) FESEM, (d) TEM and (e) HRTEM images of $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ microdumbbells. (f) HAADF-STEM image of a $\text{Fe}_2\text{O}_3\text{-TiO}_2\text{-40}$ microdumbbell with elemental mappings for Fe, Ti and corresponding overlay image.

Figure 3. FESEM images of (a) FeMOC-AmTiO₂-0, (b) FeMOC-AmTiO₂-10, (c) FeMOC-AmTiO₂-20, (d) FeMOC-AmTiO₂-40, (e) FeMOC-AmTiO₂-80 and (f) FeMOC-AmTiO₂-120.

Figure 4. Schematic illustration of the formation mechanism of FeMOC-AmTiO₂ microdumbbells synthesized with insufficient, sufficient and excessive HDA.

Figure 5. (a) J - V curves and (b) IPCEs of TiO_2 nanospheres, Fe_2O_3 microrods, Fe_2O_3 - TiO_2 -Mix and Fe_2O_3 - TiO_2 -40. (c) J - V curves and (d) IPCEs of Fe_2O_3 - TiO_2 -10, Fe_2O_3 - TiO_2 -20, Fe_2O_3 - TiO_2 -40, Fe_2O_3 - TiO_2 -80 and Fe_2O_3 - TiO_2 -120.

for Table of Content Entry

A **heterostructured Fe₂O₃-TiO₂ microdumbbell photocatalyst** is fabricated through a facile synthetic approach. With the advantages of the novel heterojunction structure, the well-defined Fe₂O₃-TiO₂ microdumbbells exhibit enhanced photoelectrochemical performance compared with Fe₂O₃ microrods, TiO₂ nanospheres and their mixture.