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Improved visible light photocatalytic activity of mesoporous FeVO₄ nanorods synthesized using a reactable ionic liquid

Hanxiang Chen ^{b,†}, Jie Zeng ^{c,†}, Mindong Chen ^b, Zhigang Chen ^c, Mengxia Ji ^a, Junze Zhao ^a,
Jiexiang Xia ^{a,*}, Huaming Li ^{a,#}

^a School of Chemistry and Chemical Engineering, Institute for Energy Research, Jiangsu University, Zhenjiang 212013, Jiangsu, China

^b School of Environmental Science and Engineering, Nanjing University of Information Science & Technology, Nanjing 210044, Jiangsu, China

^c School of the Environment and Safety Engineering, Jiangsu University, Zhenjiang 212013, Jiangsu, China

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ABSTRACT

Mesoporous FeVO₄ nanorods were successfully synthesized by calcining the precursor FeVO₄·1.1H₂O nanorods, which were obtained via a simple hydrothermal method in the presence of a reactable metal-ion-containing ionic liquid, 1-octyl-3-methylimidazolium tetrachloride ferrate(III) ([Omim]FeCl₄). The structure and morphology of the prepared samples were examined using various characterization techniques. During the synthetic process, [Omim]FeCl₄ acted as the solvent, reactant, and capping agent simultaneously. Moreover, the porous FeVO₄ nanorods as the heterogeneous photo-Fenton-like semiconductor catalyst for the degradation of tetracycline and rhodamine B under visible light irradiation exhibited excellent photocatalytic activity. This excellent photocatalytic activity of the porous FeVO₄ nanorods can be attributed to the synergistic effect of their high electron-hole pair separation rate, suitable band gap structure, and large specific surface area. The possible photocatalytic degradation mechanism of FeVO₄/H₂O₂ photocatalytic systems was also discussed in detail.

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1. Introduction

The photo-Fenton method, which is a type of advanced oxidation processes, is an efficient and widely used pretreatment method to decontaminate refractory organics such as non-biodegradable azo dyes and bio-toxic pharmaceuticals [1,2]. Ultraviolet (UV) irradiation of Fenton systems leads to the reduction of ferric ions to ferrous ions, which is always blocked in conventional Fe^{2,3+}/H₂O₂ systems, leading to the gradual deactivation of Fe²⁺ [3]. Photo-Fenton reaction systems can be

divided into two categories: homogeneous and heterogeneous photo-Fenton reaction systems. Homogeneous photo-Fenton catalysts suffer from low pH (2–3) adaptation and non-recyclable Fe sources [4–6]. Heterogeneous photo-Fenton catalysts can be recycled and are insensitive to pH, and hence have a wider range of applications [7,8]. Iron-immobilized materials, pillared interlayered clays, and Fe-based metal oxides are the three main types of heterogeneous photo-Fenton catalysts [9]. Among these catalysts, Fe-based metal oxides are the most sensitive to ultraviolet-visible (UV-Vis) light sources.

* Corresponding author. Tel/Fax: +86-511-88791108; E-mai: xjx@ujs.edu.cn

Corresponding author. Tel/Fax: +86-511-88791108; E-mai: lhm@ujs.edu.cn

† These authors contributed equally to this work.

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Hence, Fe-based metal oxides can act as photocatalysts and Fenton catalysts simultaneously, enhancing the effect of the photo-Fenton degradation reaction [9–11]. Owing to the diffusion resistance of their solid interface, heterogeneous photo-Fenton catalysts show lower H_2O_2 activation efficiency and photo-Fenton reaction rate than homogeneous photo-Fenton catalysts under similar conditions. Therefore, various heterogeneous photo-Fenton catalysts such as MIL-53(Fe) [12], urchin-like Co_3O_4 [13], and porous Fe_3O_4 nanorods [14] have been developed with nanoscale porous structures to enhance their surface areas, mass transfer processes, and pollutant degradation efficiencies. Accordingly, visible light-sensitive metal oxide semiconductors with nanoscale porous structures are promising heterogeneous photo-Fenton catalysts.

Metal oxide semiconductor photocatalysts have gained immense attention in environmental remediation owing to their interesting electrochemical properties, chemical stability, low cost, and non-toxicity [15]. FeVO_4 is an emerging visible light-driven semiconductor with suitable bandgap [16] and is widely used as a promising photocatalyst, Fenton-like catalyst [17], gas sensor material [18], and electrode material for supercapacitors [19] and Li-ion batteries [20]. Four different polymorphs of FeVO_4 (FeVO_4 -I, II, III, and IV) have been reported in the literature. Among these, the triclinic FeVO_4 -I polymorph is stable and can be prepared at room temperature, while the others are metastable phases and cannot be easily prepared under ambient conditions [21,22]. Triclinic FeVO_4 has a layered structure, which facilitates the effective separation and transfer (between layers) of photogenerated carriers, thus catalyzing the degradation reaction [23]. FeVO_4 can photocatalytically degrade various organic materials [24–26], but the performance of FeVO_4 is still far from satisfactory. This may be attributed to their limited visible-light response, fast combination of photogenerated charge carriers, and relatively low specific surface area. Various methods have been used to improve the light absorption and charge carrier separation of FeVO_4 , such as modifying with surfactants [27], constructing heterojunction photocatalysts composed of multi-semiconductors [28,29], and doping with other substances or elements [30,31]. However, there has been no report on increasing the specific surface area of FeVO_4 because the FeVO_4 particles obtained via traditional synthesis methods are prone to aggregation [16,27,32,33]. In general, compared with nonporous materials, photocatalysts with porous structures provide a large number of active sites for the degradation of organic pollutants [34–36]. Thus, the controlled synthesis of uniformly porous FeVO_4 photocatalysts with large surface areas and improved photocatalytic performance is being extensively investigated. Over the past few years, ionic liquids (ILs), as green organic salts with highly customizable structure and outstanding physicochemical properties, have gained immense attention for a wide range of applications such as the synthesis of micro/nanoscale materials [37,38]. Specifically, ILs play a vital role in the preparation of various nanostructured photocatalysts such as ultrathin $\text{Bi}_4\text{O}_5\text{I}_2$ nanosheets [39], flower-like $\text{Bi}_7\text{O}_9\text{I}_3$ nanospheres [40], BiOI hollow microspheres [41], and ZnSe hollow nanospheres [42]. Hence, the surface structure of FeVO_4 can be altered by

adding ILs to it during the synthesis process. To the best of our knowledge, the IL-assisted controlled synthesis of porous FeVO_4 has not been reported till date.

In this study, we synthesized mesoporous FeVO_4 nanorods via a two-step IL-assisted hydrothermal process using $[\text{Omim}]\text{FeCl}_4$ as the Fe source followed by calcination. $[\text{Omim}]\text{FeCl}_4$ was used as the solvent, reactant, and capping agent to restrain the aggravation of FeVO_4 grains and to achieve a uniform nanorod morphology. The mesoporous FeVO_4 nanorods, as efficient three-way photo-Fenton-like catalysts, showed lower internal resistance and higher photocatalytic activities for the degradation of tetracycline (TC) and rhodamine B (RhB) under visible light irradiation than the catalysts prepared by conventional methods. A possible three-way mechanism for the degradation of activated H_2O_2 and organic pollutants by $\text{FeVO}_4/\text{H}_2\text{O}_2$ synergetic systems was proposed.

2. Experimental

2.1. Materials

All the reagents were of analytical grade and were used without further purification. The IL, 1-octyl-3-methylimidazolium chloride ($[\text{Omim}]\text{Cl}$, 99%), was purchased from Shanghai Chengjie Chemical Co. Ltd. The metal-ion-containing IL, $[\text{Omim}]\text{FeCl}_4$, was synthesized using a method reported previously [43].

2.2. Preparation of catalysts

The porous FeVO_4 nanorods were prepared via a simple two-step method involving an IL-assisted hydrothermal process and subsequent calcination. In a typical procedure, 1 mmol of NH_4VO_3 was first dissolved in 10 mL of deionized water under magnetic stirring at 70 °C. To this solution, an aqueous solution (10 mL) of $[\text{Omim}]\text{FeCl}_4$ (with the same concentration) was added dropwise to obtain a homogeneous yellow precipitate. After stirring for 30 min, the yellow suspension was transferred to a 25 mL Teflon-sealed autoclave, which was heated at 180 °C for 24 h in an oven and then cooled to room temperature. The yellow precipitate so obtained was separated by centrifugation, washed with distilled water and absolute ethanol several times, and was dried in air at 60 °C for 8 h. The $\text{FeVO}_4 \cdot 1.1\text{H}_2\text{O}$ nanorods so obtained (labeled as $\text{FeVO}_4 \cdot 1.1\text{H}_2\text{O}$ -IL) were used as the precursor for the synthesis of porous FeVO_4 nanorods. $\text{FeVO}_4 \cdot 1.1\text{H}_2\text{O}$ -IL was calcined in a tubular furnace with N_2 atmosphere at 300, 400, and 500 °C (heating rate of 5 °C min^{-1} , cooling under ambient conditions) for 4 h and the products so obtained were labeled as $\text{FeVO}_4 \cdot 1.1\text{H}_2\text{O}$ -IL(C-300), $\text{FeVO}_4 \cdot 1.1\text{H}_2\text{O}$ -IL(C-400), and FeVO_4 -IL, respectively. For comparison, an FeVO_4 sample was also prepared using FeCl_3 as the Fe source under the same conditions (labeled as FeVO_4 -FC).

2.3. Characterization

X-ray powder diffraction (XRD) analysis was carried out on

a Bruker D8 diffractometer with high-intensity Cu-K α ($\lambda = 1.54$ Å) radiation. X-ray photoelectron spectroscopy (XPS) analysis was performed on an ESCALab MKII X-ray photo-electron spectrometer using the Mg-K α radiation. Thermogravimetric-differential scanning calorimetric (TG-DSC) analysis was carried out on an STA-449C Jupiter (NETZSCH Corporation, Germany) over the temperature range of 25–800 °C at a constant heating rate of 10 °C min⁻¹ under N₂ atmosphere. The nitrogen adsorption-desorption isotherms (obtained at -196 °C) of the catalysts were investigated using a TriStar II 3020 surface area and porosity analyzer (Micromeritics Instrument Corporation, USA). The structure of the samples was investigated using a Fourier transform spectrophotometer (FT-IR, Nexus 470, Thermo Electron Corporation) via the standard KBr disk method. Field-emission scanning electron microscopy (SEM) analysis was carried out using a field-emission scanning electron microscope (JEOL JSM-7001F) equipped with an energy-dispersive X-ray spectroscope (EDS) operating at an acceleration voltage of 10 kV. The transmission electron microscopy (TEM) images of the samples were obtained using a JEOL-JEM-2010 microscope (JEOL, Japan) operating at 200 kV. The diffuse reflectance spectra (DRS) of the samples were obtained over the wavelength range of 200–800 nm using a UV-Vis spectrophotometer (Shimadzu UV-2450, Japan). BaSO₄ was used as the reflectance standard material.

2.4. Photocatalytic activity measurement

The photocatalytic activities of the prepared FeVO₄ samples were evaluated by the degradation of TC and RhB under visible light irradiation. The experiments were carried out in a Pyrex photocatalytic reactor equipped with a 300 W Xe lamp and a UV cut-off filter ($\lambda > 400$ nm). Aeration was carried out using an air pump to ensure a constant supply of oxygen. In a typical run, 0.02 and 0.1 g of the FeVO₄ powder was dispersed into 100 mL of TC (50 mg L⁻¹) and RhB (10 mg L⁻¹) solutions, respectively. These suspensions were magnetically stirred for 30 min in the dark to establish the adsorption/desorption equilibrium. The Pyrex photocatalytic reactor was then exposed to visible light with a maximum illumination time of up to 120 min. All the experiments were performed at 30 °C using a circulating water system to prevent the thermal effects of the catalysts. During every irradiation interval (20 min), 3 mL suspension was sampled from the reactor cell and the photocatalyst powders were separated by centrifugation to obtain TC and RhB supernatant liquids, which were analyzed using a UV-Vis spectrophotometer (UV-2450, Shimadzu) at the maximal absorption wavelength (356 and 553 nm) of TC and RhB, respectively.

The experimental procedure of the photo-Fenton-like reaction was similar to the procedures mentioned above, except for the addition of 0.5 and 1 mL of H₂O₂ solutions (30 wt%) to 100 mL of TC and RhB aqueous solutions, respectively, prior to the visible-light irradiation.

2.5. Photoelectrochemical measurements

To determine the interfacial charge transfer resistance of

the prepared FeVO₄ samples, their electrochemical impedance spectroscopy (EIS) measurements were carried out using an electrochemical analyzer (CHI660B, Chen Hua Instruments, Shanghai, China) in a standard three-electrode system with a platinum wire as the counter electrode, a saturated Ag/AgCl electrode as the reference electrode, and an ITO glass as the working electrode. The FeVO₄-IL and FeVO₄-FC-modified electrodes were prepared by a simple casting method as follows. 5 mg of the as-prepared catalyst was dispersed in 0.5 mL of ethanol and EG to obtain a suspension. The resulting colloidal dispersion (20 μ L) was then dip-coated onto a 0.5 cm $\times$ 1 cm ITO glass electrode, which was dried in an oven at 55 °C for 8 h. The EIS measurements were performed in a 0.1 mol L⁻¹ KCl solution containing 5 mmol L⁻¹ Fe(CN)₆³⁻/Fe(CN)₆⁴⁻. The experiments were carried out in the absence of sunlight.

3. Results and discussion

3.1. XRD analysis

The XRD patterns of the FeVO₄-IL and FeVO₄-FC samples are shown in Fig. 1. The diffraction peaks revealed that both the samples consisted of a pure fervanite phase and showed the triclinic structure with lattice parameters $a = 6.719$ Å, $b = 8.060$ Å, and $c = 9.254$ Å, which are consistent with the values reported for triclinic FeVO₄ (JCPDS Card No. 71-1592) [44]. Both the samples showed intense diffraction peaks at $2\theta = 16.62^\circ$, 25.19° , 27.09° , and 27.79° corresponding to the (011), (012), (-201), and (1-12) lattice planes, respectively. These results are consistent with those reported previously [24,45]. No significant difference was observed in the XRD patterns of the two samples. The XRD pattern of the FeVO₄ nanorod precursor synthesized by the IL-assisted hydrothermal reaction is shown in Fig. S1 (in the Supporting Information). The diffraction peaks for this precursor could be indexed to FeVO₄·1.1H₂O [46]. In order to investigate the mechanism underlying the formation of triclinic FeVO₄-IL, FeVO₄·1.1H₂O-IL was calcined at 300, 400, and 500 °C for 4 h, and the corresponding XRD patterns are shown in Fig. S1. Both FeVO₄·1.1H₂O-IL and Fe-

Fig. 1. XRD patterns of FeVO₄-IL and FeVO₄-FC nanorods.

Fig. 2. XPS survey spectrum of FeVO₄-IL (a) and the high-resolution XPS spectra of FeVO₄-IL in the regions of Fe 2p (b), V 3p (c), and O 1s (d).

VO₄·1.1H₂O(C-300) showed two broad peaks at $2\theta = 27.94^\circ$ and 30.28° , revealing their amorphous nature. As the calcination temperature was increased to 400 °C, the crystalline phase of the FeVO₄ nanorods transformed into a transition phase, indicating that the material underwent partial amorphization prior to the complete re-crystallization. When the calcination temperature was increased to 500 °C, sharp and well-defined peaks were observed, indicating the formation of the FeVO₄ structure. These results further demonstrate that the annealing of the as-prepared amorphous fervanite (500 °C, 5 °C min⁻¹, 4 h) led to its crystallization to produce triclinic FeVO₄. This is consistent with the TG-DCS results (Fig. S2).

3.2. XPS analysis

To investigate the surface composition and chemical states of the as-prepared porous FeVO₄-IL nanorods, their XPS analysis was carried out (Fig. 2) [47–49]. The survey XPS spectrum (Fig. 2a) of the porous FeVO₄-IL nanorods showed that they were composed of Fe, V, O, and C. The carbon peak observed in the spectrum can be attributed to the presence of carbon on the surface of the sample. The Fe 2p high-resolution spectra (Fig. 2b) of the porous FeVO₄ nanorods showed Fe 2p_{3/2} and Fe 2p_{1/2} peaks at 712.12 and 725.1 eV, respectively, which are the char-

acteristic peaks of Fe³⁺ in FeVO₄-IL compounds. The V 2p XPS spectra (Fig. 2c) of the nanorods showed V 2p_{3/2} and V 2p_{1/2} peaks at 517.2 and 524.8 eV, respectively, indicating that V was present in the nanorods in the V⁵⁺ state [50]. Meanwhile, the high-resolution spectrum (Fig. 2d) of the nanorods showed an O 1s peak at 530.21 eV corresponding to the O²⁻ state of O atoms [45]. These results demonstrate that pure porous FeVO₄-IL nanorods were successfully synthesized.

Fig. 3. FT-IR spectra of FeVO₄-IL and FeVO₄-FC nanorods.

3.3. FT-IR analysis

The composition and functional groups of the as-synthesized $\text{FeVO}_4\text{-IL}$ and $\text{FeVO}_4\text{-FC}$ samples were further examined by FT-IR spectroscopy and the results are shown in Fig. 3. The samples showed similar infrared characteristic peaks. Both the samples showed shoulder bands (ca. 970 cm^{-1}) corresponding to the symmetric stretching mode of the VO_4 groups. The samples showed three dominant bands at ca. 914, 838, and 666 cm^{-1} corresponding to the split asymmetric stretching mode of the VO_4 groups. The weak bands (ca. 751 cm^{-1}) could be attributed to the $\text{V-O} \cdots \text{Fe}$ bridging stretching. The bands at 509 cm^{-1} can be attributed to the V-O-V deformation mode, which was strongly mixed with the Fe-O stretching of octahedral Fe-O . These results are consistent with those reported previously [50]. In addition, $\text{FeVO}_4\text{-FC}$ showed a weak band at around 3398 cm^{-1} and a small band at 1612 cm^{-1} corresponding to the O-H stretching and O-H bending modes, respectively [51]. No characteristic peaks of imidazolium C-H stretching (of the IL) were observed between 3000 and 3200 cm^{-1} in the FT-IR spectra. Hence, it can be stated that washing the samples with deionized water and alcohol resulted in the complete removal of the IL from their surface. The FT-IR spectra of the $\text{FeVO}_4\cdot 1.1\text{H}_2\text{O-IL}$ samples are shown in Fig. S3.

3.4. SEM, TEM, and EDS analysis

The morphology and microstructure of the as-synthesized $\text{FeVO}_4\cdot 1.1\text{H}_2\text{O-IL}$ and $\text{FeVO}_4\text{-IL}$ samples were further investigated by SEM and TEM analyses. As shown in Fig. 4a, the $\text{FeVO}_4\cdot 1.1\text{H}_2\text{O-IL}$ nanorods were uniform and smooth with

Fig. 4. (a) High magnification SEM images of $\text{FeVO}_4\cdot 1.1\text{H}_2\text{O}$ nanorods (b, c) SEM, (d) TEM, (e) HRTEM images, and (f) EDX spectrum of porous $\text{FeVO}_4\text{-IL}$ nanorods.

lengths and diameters of $2\text{--}3\text{ }\mu\text{m}$ and $50\text{--}100\text{ nm}$, respectively. The morphology of these nanorods was similar to that of $\text{FeVO}_4\text{-IL}$ (Figs. 4b and 4c), except for the slightly rough surface of the calcined nanorods [25,44]. $[\text{O}(\text{mim})]^+$ ions have long alkyl carbon chains and facilitate the directional growth of pre-organized structures by preventing or delaying the crystal growth, thus promoting anisotropy ultimately [37]. Meanwhile, chloride ions as capping agents for some crystal surfaces affect their morphology by controlling the growth rate. The TEM image of $\text{FeVO}_4\text{-IL}$ (Fig. 4d) showed that it consisted of pores with diameters ranging from 5 to 20 nm . The porous structure of the sample can be attributed to the dehydration and recrystallization during the annealing treatment. Moreover, the high-resolution TEM image of $\text{FeVO}_4\text{-IL}$ (Fig. 4e) showed that its interplanar distance was 0.505 nm , which is consistent with the d -spacing of the (011) planes of triclinic FeVO_4 . The EDS analysis of the nanorods (Fig. 4f) showed the presence of Fe, V, O, and Si in them. The peak corresponding to Si can be attributed to the Si substrate. The Fe/V molar ratio was approximately 1:1, further confirming that the porous nanorod samples were composed of the FeVO_4 crystalline phase. This is consistent with the XRD, XPS, and FT-IR analysis results.

3.5. BET analysis

In order to investigate the surface properties of the porous $\text{FeVO}_4\text{-IL}$ nanorods, their Brunauer-Emmett-Teller (BET) specific surface areas and the corresponding Barrett-Joyner-Halenda pore size distributions were investigated by carrying out nitrogen adsorption-desorption experiments. Fig. 5a shows the nitrogen absorption-desorption isotherms of the porous $\text{FeVO}_4\text{-IL}$ nanorods. The nanorods showed type-IV isotherms (BDDT classification) with a hysteresis loop, suggesting the presence of mesopores in them [52]. The BET specific surface area of the $\text{FeVO}_4\text{-IL}$ nanorods was calculated to be $255.8294\text{ m}^2/\text{g}$. The pore size distribution curve (inset in Fig. 5a) of the $\text{FeVO}_4\text{-IL}$ nanorods showed that their average pore diameter was 3.6 nm . These mesopores were generated by the dehydration of the precursors during the calcination process. These results are consistent with the TEM results. The porous structure of the $\text{FeVO}_4\text{-IL}$ nanorods provided adequate number of active and adsorption sites for the substrate molecules during the photocatalytic reaction.

3.6. Optical properties

The absorption and utilization of solar light by semiconductors is determined by their optical properties [53,54]. The optical properties of the porous $\text{FeVO}_4\text{-IL}$ and $\text{FeVO}_4\text{-FC}$ nanorods were evaluated by examining their UV-Vis DRS results (Fig. 5b), which revealed that the onset absorption edge of $\text{FeVO}_4\text{-IL}$ and $\text{FeVO}_4\text{-FC}$ were 560 and 580 nm , respectively. The slightly broader absorption boundary of $\text{FeVO}_4\text{-FC}$ can be attributed to its orange-yellow color, which is darker than the neon yellow color of $\text{FeVO}_4\text{-IL}$. Hence, it can be stated that the microstructure and surface properties of ferric vanadates can be controlled and regulated by $[\text{O}(\text{mim})]\text{FeCl}_4$, thus enhancing their

Fig. 5. (a) Nitrogen absorption-desorption isotherm of porous FeVO₄-IL nanorods; (b) UV-vis diffuse reflectance spectra of FeVO₄-IL and FeVO₄-FC nanorods.

Fig. 6. Photo-Fenton-like photodegradation of TC (a) and RhB (b) with or without the presence of FeVO₄-IL, FeVO₄-FC, H₂O₂ and visible light irradiation.

photocatalytic efficiency.

3.7. Photocatalytic activity of the prepared samples

As shown in Fig. 6a, the photocatalytic degradation performance of FeVO₄-FC and the porous FeVO₄-IL nanorods was evaluated by carrying out the degradation of TC under visible light irradiation. After irradiation for 120 min, TC showed negligible self-degradation. Moreover, when H₂O₂ and FeVO₄-IL were separately employed for the photocatalytic degradation of TC, the removal of TC was less than 10%. During the same irradiation time, the FeVO₄-IL/H₂O₂ system showed higher degradation efficiency (79%) than the FeVO₄-FC/H₂O₂ photo-Fenton-like system (66%). When the Xe lamp was turned off, the TC removal efficiency of FeVO₄-IL/H₂O₂ reduced to around 9%. These results suggest that owing to the presence of Fe(III) and V(V), both FeVO₄-FC and FeVO₄-IL activated H₂O₂ to generate •OH radicals [32], which are the main active species responsible for the decomposition of TC in photo-Fenton-like systems. In addition, FeVO₄ as a semiconductor produced electron-hole pairs under illumination, and the valence band (VB) holes exhibited a strong oxidative potential to oxidize and degrade the adsorbed TC molecules. Furthermore, Fe(III) bonded

strongly with the multiple O- and N-containing moieties of TC and acted as a photoreactive species in the TC photodegradation process. The porous FeVO₄-IL nanorods were more active than FeVO₄-FC in all the three processes mentioned above. As a result, the porous FeVO₄-IL nanorods, as a heterogeneous photo-Fenton-like catalyst, showed good photocatalytic activity towards the degradation of TC under visible light irradiation.

The photocatalytic activities of the FeVO₄-FC and porous FeVO₄-IL nanorod samples were further measured by the photodegradation of RhB under different conditions. As shown in Fig. 6b, RhB showed negligible self-degradation after 120 min. When FeVO₄-IL and H₂O₂ were used separately for the photocatalytic degradation of RhB, their RhB removal efficiencies were 8% and 47%, respectively. However, the FeVO₄-IL/H₂O₂ photo-Fenton-like system exhibited a RhB removal efficiency of up to 89% during the same irradiation time. When the Xe lamp was turned off, the Fenton-like catalytic degradation of RhB was reduced by 19%. The FeVO₄-FC/H₂O₂ photo-Fenton-like system showed a poor RhB degradation efficiency (64%) during the same irradiation time.

Furthermore, the photocatalytic stability of the porous FeVO₄-IL nanorods was investigated by carrying out repeated RhB degradation experiments, which was also very important

Fig. 7. (a) Cycling runs of porous FeVO₄-IL nanorods under visible light irradiation for 2 h; (b) XRD patterns of porous FeVO₄-IL nanorods after the cycling photocatalytic experiments.

from a practical point of view. The porous FeVO₄-IL nanorods showed no significant loss of photocatalytic activity after the repeated degradation experiments (Fig. 7a), suggesting that they were highly stable. The XRD pattern of the recycled photocatalyst (Fig. 7b) also confirmed that the structure of the photocatalyst did not change after recycling. These results suggest that porous FeVO₄-IL nanorods with good structural stability and photocatalytic performance are effective photo-Fenton-like catalysts for the degradation of organic compounds.

Hydroxyl radicals have an oxidative ability high enough to attack many organic molecules, and hence are dominant active species in photo-Fenton-like processes [2]. Therefore, in this study, we used isopropanol in the trapping experiments as an effective scavenger for hydroxyl radicals. As shown in Fig. 8, when isopropanol was added to the photo-Fenton-like system, its RhB degradation efficiency decreased significantly (as expected) and only 20% of RhB was photodegraded eventually. Moreover, EDTA-2Na was used in the hole (h⁺) trapping experiments. The RhB photodegradation rate of the FeVO₄/H₂O₂ system decreased significantly in the presence of EDTA-2Na, suggesting the presence of photogenerated holes (h⁺) in the system for the degradation of the pollutants. This can be explained by the fact that FeVO₄, which is an n-type semiconduc-

tor, can be excited by visible light irradiation, thus generating electron-hole pairs, and most of the transition electrons in the conduction band (CB) are consumed by H₂O₂, Fe³⁺, and V⁵⁺ to generate •OH radicals, Fe²⁺, and V⁴⁺, respectively. This leaves a certain amount of photogenerated holes in the VB to oxidize and decompose the adsorbed RhB molecules or to recombine with electrons. Hence, the synergistic combination of semiconductors and Fenton-like systems shows high electron-hole separation rate and highly activated H₂O₂.

The transition efficiency of photogenerated charge carriers determines the photodegradation ability of semiconducting materials [55–57]. Accordingly, the EIS measurements of both the FeVO₄-FC and FeVO₄-IL photocatalysts (Fig. 9) were carried out to determine the interfacial charge transfer resistance of the ITO/FeVO₄-IL and ITO/FeVO₄-FC films. It can be observed clearly from the figure that the ITO/FeVO₄-IL film showed a much lower resistance (and hence higher electrical conductivity) than the ITO/FeVO₄-FC film. This indicates that FeVO₄-IL exhibited effective separation of the photogenerated electrons and holes [58]. Hence, on the basis of these results and those obtained from the DRS analysis, it can be stated that [Omim]FeCl₄ (IL) can control and regulate the microstructure and surface properties of ferric vanadates, thus enhancing their photocatalytic reactivity.

Fig. 8. Photo-Fenton-like catalytic activities of the porous FeVO₄-IL nanorod catalyst for the degradation of RhB with or without adding isopropanol and EDTA-2Na under visible light irradiation.

Fig. 9. Comparison of electrochemical impedance spectroscopy (EIS) between FeVO₄-IL and FeVO₄-FC samples.

Fig. 10. $(\alpha E_{\text{photon}})^2$ vs. E_{photon} curves (a) and valence band spectrum (b) of porous FeVO₄-IL nanorods.

3.8. Photocatalytic degradation mechanism of semiconductor photo-Fenton-like synergetic systems

The band gap energy of semiconductor materials determines their sunlight absorption capacity. Typically, the optical absorption of crystalline semiconductors near the band edge is given by the following equation: $\alpha E_{\text{photon}} = K(E_{\text{photon}} - E_g)^{n/2}$, where K is a constant, α is the absorption coefficient, E_{photon} is the discrete photo energy, and E_g is the bandgap energy. The value of n depends on the transition characteristics of the semiconductor (direct transition $n = 1$ and indirect transition $n = 4$) [59,11]. Because FeVO₄ is a direct-transition semiconductor, its E_g value is determined by the energy intercept of the $(\alpha E_{\text{photon}})^2$ vs. E_{photon} curve. The DRS analysis results showed that the absorption edge of FeVO₄ was at 560 nm (because of the direct transition) and the corresponding band-gap energy was 2.25 eV (Fig. 10a). These results suggest that the FeVO₄-IL samples had an optimum bandgap for visible light activation for photocatalytic decomposition of organic contaminants. Furthermore, the VB potentials E_{VB} of the FeVO₄-IL samples were determined from their VB XPS spectra (Fig. 10b), and the CB potentials E_{CB} were determined using the formula: $E_{\text{CB}} = E_{\text{VB}} - E_g$ [60,61]. Consequently, the estimated VB and CB positions of the porous

FeVO₄-IL nanorods were 2.18 and -0.17 eV, respectively. Because the $E^0(\text{O}_2/\bullet\text{O}_2^-)$ of the FeVO₄-IL samples was -0.046 eV vs. NHE, the activation of the electrons transitioned and accumulated on their CB (-0.17 eV vs. NHE) was sufficient to reduce O_2 to generate $\bullet\text{O}_2^-$. Hence, it can be stated that $\bullet\text{O}_2^-$ was also the active species during the photodegradation process.

On the basis of the results discussed thus far, a possible mechanism for the photodegradation of TC by the FeVO₄-IL/H₂O₂ photo-Fenton-like system was proposed (Fig. 11). Owing to the large specific surface area of the porous FeVO₄-IL nanorods, a large number of TC molecules were adsorbed on their surface in the dark. Upon visible light irradiation, the photo-excited electrons transitioned from the VB to the CB, leaving an equal number of holes to oxidize the adsorbed TC molecules. Meanwhile, the adsorbed O_2 molecules, Fe³⁺ ions, and V⁵⁺ ions on the surface of the porous FeVO₄-IL nanorods were reduced to $\bullet\text{O}_2^-$, Fe²⁺, and V⁴⁺ ions, respectively, by the photogenerated electrons [27]. Furthermore, both the reduced Fe²⁺ and V⁴⁺ ions on the surface of the porous FeVO₄-IL nanorods accelerated the decomposition of H₂O₂ in the solution [32], generating oxidative $\bullet\text{OH}$ radicals during the Fenton reaction. Fe²⁺ and V⁴⁺ ions were then oxidized to Fe³⁺ and V⁵⁺ ions, respectively, by H₂O₂, closing the cycle loop. Moreover, some H₂O₂ was also reduced by the photogenerated electrons to release $\bullet\text{OH}$ radicals. Finally, the $\bullet\text{O}_2^-$ and $\bullet\text{OH}$ radicals obtained during the three processes mentioned above attacked the adsorbed TC molecules on the surface of the porous FeVO₄-IL nanorods and degraded them to CO₂ and H₂O.

4. Conclusions

The controlled synthesis of porous FeVO₄-IL nanorods was carried out successfully via an IL-assisted hydrothermal method followed by calcination. The physicochemical characterization results showed that pure porous FeVO₄-IL nanorods with large specific surface areas were successfully synthesized. Moreover, the photo-Fenton-like degradation activities of the porous FeVO₄-IL and FeVO₄-FC samples were evaluated by carrying out the degradation of TC by them under visible light irradiation. The porous FeVO₄-IL nanorods showed higher rate constant and TC degradation efficiency than FeVO₄-FC. This

Fig. 11. A schematic illustration of organics degradation over photo-Fenton-like porous FeVO₄-IL nanorod catalyst under visible light irradiation.

suggests that [Omim]FeCl₄ played an important role during the synthesis of the FeVO₄-IL nanorods and significantly enhanced their photocatalytic degradation performance. The excellent photocatalytic activity of the porous FeVO₄ nanorods can be attributed to their high electron-hole separation rate, suitable band gap structure, and large specific surface area. Meanwhile, the satisfactory efficiency of the FeVO₄-IL/H₂O₂ system for the degradation of RhB and TC can be attributed to the synergistic effect of FeVO₄-IL and H₂O₂.

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Graphical Abstract

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Improved visible light photocatalytic activity of mesoporous FeVO₄ nanorods synthesized using a reactable ionic liquid

Hanxiang Chen, Jie Zeng, Mindong Chen, Zhigang Chen, Mengxia Ji, Junze Zhao, Jiexiang Xia*, Huaming Li*
Jiangsu University; Nanjing University of Information Science & Technology

A mesoporous FeVO₄ nanorod photocatalyst with the pore diameter of 5–20 nm was synthesized by an Fe-based ionic liquid-assisted method. The porous structure of the photocatalyst provided a large number of adsorption and reactive sites for photodegradation.

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反应型离子液体辅助合成钒酸铁介孔纳米棒及其增强可见光光催化活性

陈翰祥^{b,†}, 曾洁^{c,†}, 陈敏东^b, 陈志刚^c, 季梦夏^a, 赵君泽^a, 夏杰祥^{a,*}, 李华明^{a,#}

^a江苏大学化学化工学院, 能源研究院, 江苏镇江212013

^b南京信息工程大学环境科学与工程学院, 江苏南京210044

^c江苏大学环境安全与工程学院, 江苏镇江212013

摘要:近年来, 环境污染与能源短缺已经成为人类需解决的问题, 因此新型绿色能源的开发显得尤为重要。在众多新型能源当中, 太阳能由于其安全无害、无二次污染、应用前景广泛等优点而备受关注。半导体光催化技术作为一项可以直接将太阳能转化为化学能的新兴技术, 可以有效地利用太阳光实现环境治理和能源转化的目的, 已被应用于光催化分解水、光催化合成氨、光催化二氧化碳还原以及光催化降解有机污染物等不同研究领域。然而传统的光催化剂材料TiO₂对太阳光的利用效率较低, 大大限制了光催化技术的广泛应用。因此, 研发新型高效光催化半导体材料成为人们的研究热点。

相比于普通的体材料, 低维和小尺寸纳米材料往往具备更为优良的物化特性。一维尺寸的三元钒酸盐材料作为一类极具前景的多功能纳米材料, 在光学设备、光催化降解、电极材料以及电化学传感器等诸多领域都具有广泛应用。其中, 钒酸铁材料作为钒酸盐系列中的一员, 其有着合适的带隙且能响应可见光, 是一种具有研究前景的光催化材料。三斜相的钒酸铁具有层状结构, 这有利于光生载流子在层间进行有效的分离和迁移, 从而提高光催化降解性能。同时, 离子液体作为一种结构高度可调的绿色有机盐, 在微纳米材料的可控制备方面起着关键作用。本文选取1-辛基-3-甲基咪唑氯盐作为反应铁源, 利用离子液体辅助溶剂热法合成了钒酸铁前驱体材料FeVO₄·1.1H₂O。通过调控煅烧温度, 可控制备了尺寸均一的介孔钒酸铁纳米棒材料。同时, 选取无机盐氯化铁作反应铁源制备了钒酸铁纳米棒作为对比。

根据X射线粉末衍射图谱可知, 当煅烧温度升到400 °C时, 前驱体材料的晶相转变为过渡相; 当煅烧温度升到500 °C时, 出现了清晰的归属于钒酸铁的特征衍射峰, 表明钒酸铁结构形成。从扫描电镜图可以清楚地观察到所制备的前驱体材料为结构均一且表面光滑的纳米棒结构, 其长度为2–3 μm。经过煅烧处理后, 在钒酸铁纳米棒表面形成孔径为5–20 nm的介孔结构, 这可能是由于煅烧过程中前驱体材料发生脱水重结晶。结合X射线衍射图谱, 确定了介孔钒酸铁纳米棒的形成过程。此外, 通过氮气吸附-脱附等温线得到了介孔钒酸铁材料的比表面积。在光催化降解过程中, 大的催化剂比表面积可以为反应基质提供充分的吸附位点和反应活性位点, 从而有利于提高光催化反应活性。

选取抗生素四环素作为目标污染物分别考察了在无机盐(FeVO₄-FC)和离子液体(FeVO₄-IL)条件下制备的钒酸铁材料的催化性能。其中, 四环素的自降解作用可以忽略。在加入H₂O₂光照120 min后, FeVO₄-IL表现出比FeVO₄-FC更高的光催

化性能. 此外, 采用染料罗丹明B进一步确定所制备材料的光催化性能. 结果表明, 在相同的光照时间后, $\text{FeVO}_4\text{-IL}$ 有着更高的催化降解活性. 对介孔纳米棒进行了稳定性测试, 在四次循环后, 未发现其光催化活性有明显降低, 其结构也保持不变. 电化学阻抗测试结果显示, 相比于 $\text{FeVO}_4\text{-FC}$ 材料, $\text{FeVO}_4\text{-IL}$ 有着更小的阻值, 表明离子液体可控合成的介孔纳米棒材料更有利于光生电荷的传输, 从而增强了光催化降解活性. 基于一系列表征结果, 我们提出了多孔钒酸铁纳米棒可能的光催化降解机制.

关键词: 钒酸铁; 可见光; 光催化降解; 离子液体

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*通讯联系人. 电话: (511)88791108; 传真: (511)88791108; 电子信箱: xjx@ujs.edu.cn

#通讯联系人. 电话: (511)88791108; 传真: (511)88791108; 电子信箱: lhm@ujs.edu.cn

†共同第一作者.

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