Graphical Abstract

Photocatalytic conversion of nitrite in aqueous solution over nanocomposite photocatalyst $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ using different photosources

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A B S T R A C T

Three crystal phases of BiPO_4 (HBIP, nMBIP and mMBIP), corresponding photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$) and their nanocomposite photocatalyst ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$) were prepared by hydrothermal, ultrasonic dispersion and liquid boiling methods. The prepared photocatalysts were characterized by X-ray diffractometer (XRD), energy dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM) and UV-vis diffuse reflectance spectra (DRS). The catalytic activity of prepared photocatalysts was evaluated via photocatalytic conversion rate of nitrite under ultraviolet-light, visible-light and simulated solar-light irradiations. These three photocatalysts all exhibited excellent performance under simulated solar-light irradiation and reached 85.36 %, 84.42 % and 78.53 % conversion rates, respectively, for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$. Particularly, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ was also found to play a high catalytic activity, resulting in 78.68 %, 70.04 % and 82.57 % conversion rates, respectively, under ultraviolet-light, visible-light and simulated solar-light irradiation. In addition, the study of used times showed that the prepared photocatalysts can be

effectively recycled without an apparent inactivation on the photocatalytic activity. This research may provide a potential way for converting nitrite and other contaminants by utilizing solar energy efficiently.

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Keywords: Photocatalytic conversion, Nitrite, BiPO_4 , Up-conversion luminescence agent ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$), Different photosources

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1. Introduction

In nature, the nitrite anions (NO_2^-) often form in the process of ammonia (NH_3) oxidation by nitrite bacteria and is also produced as a comparatively stable intermediate from nitrate ion reduction [1-3]. With the rapid development of modern industry and agriculture, the concentration of nitrite anion in water body has been significantly rising due to excessive discharge of industrial effluents, overusing nitrogenous fertilizer and incomplete abiotic denitrification process [4-6]. At present, the nitrite has been considered as a common inorganic environmental contaminant, which presents a serious toxicity to human health [7-9]. The excess intake of nitrite, which combine with hemoglobin in the blood to form methemoglobin in the body, as a result, can cause human poisoning [10]. Further,

nitrite can be converted into carcinogenic nitrosamine that is known to cause hypertension, stomach and bowel cancers, leukemia, and brain tumors [11-13]. Thus, the World Health Organization recommends the maximum nitrite concentration of 3.0 mg/L in drinking water to avoid health risks [14]. And as a result, it is very necessary for us to remove the nitrite anion in drinking water in order to satisfy the requirement of human health.

In principle, the conventional technologies for nitrite conversion have contained physical, micbiological and chemical methods [15-17]. Physical methods that including ionic membrane, reverse osmosis and electron dialysis usually present the advantage to minimize nitrite concentration in a large scale. However, these methods often generate high-cost issues due to specific requirements for supervision, maintenance, and secondary treatment [18]. Micbiological methods often use nitrite as sources of oxygen in anaerobic conditions or as sources of nitrogen as nutrient in aerobic conditions. These methods are able to treat a great deal of sewage, but the process is relatively slow and complex. Chemical methods are advantageous to destroy or transform nitrite into a less toxic nitrogen form, and the catalytically reaction process is efficient [19]. Among chemical methods, photocatalysis is particularly suitable for the conversion of nitrite because of its simple and environmentally friendly process by utilizing the clean, inexhaustible and easily accessible resources of solar energy [20-22].

In recent years, various kinds of semiconductors used to photocatalytic reaction have received the widespread attentions. As a ideal photocatalyst, some important factors must be accounted for. The number one is that the used photocatalyst can effectively remove the pollutants in water directly using its absorption light. Another is the easy separation of photocatalyst from treated water. In addition, the photocatalyst can be reused effectively. For a long time, titanium dioxide (TiO_2) has been widely investigated in the field of photocatalysis due to its relatively high catalytic activity, physical and

chemical stability, avirulence and inexpensiveness [23-25]. However, in order to convert nitrite into a less toxic nitrogen form, semiconductor TiO_2 are not quite suitable somewhat because of the limitation of the absorption edge and the recombination of photogenerated charges. What we need is a photocatalyst which has the appropriate wide band gap, at the same time more negative reduction potential (conduction band (CB)) and more positive oxidation potential (valence band (VB)) [26,27].

Based on the investigation, it was considered that the bismuth phosphate (BiPO_4) can better satisfy the requirements above. BiPO_4 is usually used in the photocatalytic conversion of organic contaminant, this research reported the photocatalytic conversion of inorganic ion for the first time. It was noted that BiPO_4 exhibited three main crystal phases: hexagonal BiPO_4 (space group: P3_121 , HBIP), monoclinic BiPO_4 (space group: $\text{P2}_1/\text{n}$, nMBIP) and monoclinic BiPO_4 (space group: $\text{P2}_1/\text{m}$, mMBIP) [28]. These three crystal phases of BiPO_4 have different 3D networks made up of PO_4 tetrahedral and BiO_8 polyhedral, so they have been proved to display different catalytic activities [29]. Among the three crystal phases of BiPO_4 , the superior catalytic performance of nMBIP may be ascribed to its most distortion PO_4 tetrahedron causing slightly wide light response range [28]. However, as a wide band-gap semiconductor the BiPO_4 powder is active only under ultraviolet-light excitation, which accounts for less than 5.0 % of the entire solar spectrum [30]. Therefore, it is too inefficient to harness solar energy by only using pure BiPO_4 . Some up-conversion luminescence agent can be used as an alternative, because they are able to emit photons of ultraviolet-light under visible-light excitation [31].

Recently, one of up-conversion luminescence agents, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, has been widely used in many fields due to its high conversion luminescence efficiency and chemical stability [32]. Therefore, we prepared $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ as an effective up-conversion luminescence agent to combine with BiPO_4 forming visible-light responding photocatalyst.

In this article, we prepared three crystal phases of BiPO_4 and corresponding nanocomposite photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$). Considering their different band gaps causing various absorption edges, matching different emission peaks of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ was designed to effectively utilize the ultraviolet-lights resulted in up-conversion luminescence of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ at the most extent. The influence of different photosources on the performance of four nanocomposite photocatalysts was investigated through the photocatalytic conversion rate of nitrite. In addition, the effects of simulated solar-light irradiation time and used times on the photocatalytic conversion rate of nitrite were also studied. In the end, the possible mechanism on simulated solar-light photocatalytic conversion of nitrite in the presence of nanocomposite photocatalysts were proposed. The experimental results showed that the prepared photocatalysts all displayed quite high performance in the photocatalytic conversion of nitrite. Furthermore, it can be predicted that the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ can more effectively utilize solar light to perform the photocatalytic conversion of nitrite.

2. Experimental

2.1. Materials and reagents

Bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, 98 %, Sinopharm Chemical Reagent Co., Ltd, China) and phosphoric acid (H_3PO_4 , 85 %, Sinopharm Chemical Reagent Co., Ltd, China) and were used to synthesize the BiPO_4 particles. Erbium oxide (Er_2O_3 , 99.999 %, Sinopharm Chemical Reagent Co., Ltd, China), yttrium oxide (Y_2O_3 , 99.999 %, Aladdin Chemical Co., Ltd, China), aluminum nitrate nonahydrate ($(\text{Al}(\text{NO}_3)_3) \cdot 9\text{H}_2\text{O}$, analytical pure, Sinopharm Chemical Reagent Co., Ltd, China), citric acid ($\text{C}_6\text{H}_8\text{O}_7$, analytical pure, Sinopharm Chemical Reagent Co., Ltd, China) and nitric acid

(HNO₃, guaranteed pure, Aladdin Chemical Co., Ltd, China) were used to synthesize the up-conversion luminescence agent (Er³⁺:Y₃Al₅O₁₂). Sodium nitrite (NaNO₂, 99 %, Aladdin Chemical Co., Ltd, China) was used to make stock and standard solution of NO₂⁻. All chemicals were used without further purification.

2.2. Preparation of Er³⁺:Y₃Al₅O₁₂/BiPO₄ nanocomposite photocatalysts

2.2.1. Synthesis of Er:Y₃Al₅O₁₂ as up-conversion luminescence agent

Er³⁺:Y₃Al₅O₁₂, as the up-conversion luminescence agent, was synthesized by sol-gel and calcination methods [33]. Dissolve stoichiometric Y₂O₃ (2.2715 g) and Er₂O₃ (0.0128 g) into right quantity of hot HNO₃ aqueous solution (about 60 °C) to prepare Y(NO₃)₃ and Er(NO₃)₃ solutions. The obtained solutions were mixed with Al(NO₃)₃·9H₂O (12.6208 g) with magnetic stirring up to the homogeneous and transparent solution was obtained. Then C₆H₈O₇ (33.9351 g) was added into the above mixture solution (mol ratio of citric acid: metal ion is 3:1) and was further stirred and heated at 50-60 °C until the transparent sol was formed. And then transfer the transparent sol into a electrothermal blowing dry box. After heated at 80 °C for 24 h and then cooled down in the air, the obtained xerogel was ground into fine homogeneous powders. Subsequently, the particles were heated at 1100 °C and kept for 2.0 h so that residual organic components and nitrate ions can be removed completely. Finally, grind down the sintered substance into uniform powders after taking it out of the muffle furnace and cooling down to the room temperature naturally. After carefully grinding, the desired white Er³⁺:Y₃Al₅O₁₂ particles were obtained.

2.2.2. Preparation of BiPO₄

Three crystal phases of BiPO₄ powders were synthesized through different hydrothermal

processes [34]. Firstly, 18 mmol $\text{Bi}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (8.731 g) and 180 mL H_3PO_4 (12 M) were put into a beaker while stirring magnetically for 1.0 h to form a homogeneous solution. Subsequently, the obtained suspension was transferred into three Teflon-lined stain-less steel autoclaves and the temperatures were maintained at 20 °C, 100 °C and 200 °C, respectively, without shaking or stirring. After 72 h hydrothermal treatment and cooling down to room temperature, the three products (hexagon BiPO_4 (space group: P3_121 , HBIP), monoclinic BiPO_4 (space group: P21/n , nMBIP) and monoclinic BiPO_4 (space group: P21/m , mMBIP)) were obtained, respectively, by filtering off, washing with distilled water for several times, and being dried at 80 °C for 24 h [28].

2.2.3. Preparation of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ nanocomposite photocatalysts

The corresponding photocatalysts and their nanocomposite photocatalyst were prepared through the ultrasonic dispersion and liquid boiling methods [35]. The $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, three crystal phases of BiPO_4 powders and the mixture of three crystal phases of BiPO_4 were mixed as the 0.3:1.0 mass ratios of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and BiPO_4 , respectively. Then, each of the mixture was added into a beaker filled with 30 mL deionizer water and then fully dispersed by ultrasound of 80 kHz frequency and 50 W output power for 30 min. The well-suspended liquid was heated to boiling point for 30 min with constantly magnetic stirring. After filtration and lavation, the separated deposit was put into crucibles and sintered in a muffle furnace at 300 °C for 120 min. At last, the four photocatalysts were obtained successfully.

2.3. Characterization of prepared photocatalysts

The prepared samples were characterized by X-ray diffraction (XRD) on Bruker-axs D-8 X-ray diffractometer using Ni filtered $\text{Cu K}\alpha$ radiation in the range of 2θ from 10° to 70°. Morphologies and

structures of the prepared samples were further examined with JEOL JSM-5610LV electron scanning microscope (SEM). The element type and content were analyzed by energy dispersive X-ray spectroscopy (EDX) on JEOL JSM-5610LV. UV-vis diffuse reflectance spectra (DRS) of samples were measured by using SHIMADZU-UV-3600 spectrophotometer. Fourier transform infrared (FT-IR) spectra of the samples were performed on a Nicolet 5700 FT-IR spectrometer. Experiment operations were conducted in a photochemical reaction instrument (TL-GHX-V, Xujiang Electromechanical Plant, China). Concentrations of NO_2^- were determined in the liquid phase with Vian ProStar-210.

2.4. Experiments of nitrite photocatalytic conversion

The photocatalytic conversion experiments of nitrite were carried out by using a photochemical reaction instrument, in which nitrite aqueous solutions (50 mL and 10 ppm) containing a specified amount of photocatalyst (50 mg) were put in a test tube. Before light irradiation, the suspension was magnetically and constantly stirred in the dark for 30 min to reach the adsorption-desorption equilibrium. Then the suspension was irradiated for 8.0 h by using 500 W xenon lamp, 500 W mercury lamp and 500 W halogen lamp as simulated solar light source, ultraviolet-light source and visible-light source, respectively. At interval of 2.0 h, 5.00 mL solutions were sampled and centrifuged to remove the photocatalysts. The photocatalytic conversion rate was determined by $(1 - C_t/C_0) \times 100\%$ formula, in which C_0 and C_t were the initial concentration of nitrite and the instant concentration of nitrite at irradiation time (t).

3. Results and discussion

3.1. XRD, EDX, SEM, UV-DRS and FT-IR of prepared samples

The prepared samples were firstly characterized by using XRD measurements. As shown in Fig. 1(a), the synthesized $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ was identified according to JCPDS 33-0040 file of $\text{Y}_3\text{Al}_5\text{O}_{12}$ at $2\theta = 18.15^\circ$ (211), 33.50° (400), 41.50° (422), 46.62° (521) and 55.12° (532). It displays that in the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ the $\text{Y}_3\text{Al}_5\text{O}_{12}$ has formed, in which the Er^{3+} ions entered the crystal lattice replacing the partial Y^{3+} ions. In Fig. 1(b-1), the characteristic diffraction peaks at $2\theta = 19.1^\circ$, $2\theta = 21.5^\circ$ and $2\theta = 29.5^\circ$ were marked as (100), (101) and (200) planes of HBIP (hexagon BiPO_4 (space group: P3_121 , HBIP)) in accordance with the standard pattern (JCPDS 45-1370). In Fig. 1(c-1), the sample exhibits feature peaks at $2\theta = 17.0^\circ$, 21.4° and 34.5° attributed to the planes (-101), (-111) and (-202) of nMBIP (monoclinic BiPO_4 (space group: P21/n , nMBIP)) as identified using the standard data JCPDS 80-0209. In Fig. 1(d-1), the main peak positions appear at $2\theta = 18.5^\circ$, 22.8° and 37.0° , corresponded to the (100), (011) and (200) planes, were successfully indexed to mMBIP (monoclinic BiPO_4 (space group: P21/m , mMBIP)) and consistent with JCPDS 43-0637. In Fig. 1(b-2, c-2, d-2 and e), the XRD patterns of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ were given, respectively. The main characteristic diffraction peaks of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and three different crystal phases of BiPO_4 can be found, which demonstrates that the crystal conformations of BiPO_4 and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ are not changed in the respective $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ nanocomposite particle.

Fig. 1.

The SEM images of prepared samples are all given in Fig. 2. From the image of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ depicted in Fig. 2(a), numerous irregular spherical particles with an average size of 40 - 50 nm can be seen, which should be identified as the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$. And that, for these three crystal phases of BiPO_4

they display different sizes and shapes indeed. As shown in Fig. 2(b-1), as schistose crystals the hexagon BiPO_4 (space group: $P3_121$, HBIP) give 1.0 - 1.5 μm length, 100 - 200 nm width and 30 - 50 nm height. In Fig. 2(c-1), it can be found that the prepared monoclinic BiPO_4 (space group: $P21/n$, nMBIP) are some regular cubic nanoparticles with size about 50 - 100 nm. In Fig. 2(d-1), the monoclinic BiPO_4 (space group: $P21/m$, mMBIP) particles present cuboid structure, which are 1.5 - 2.0 μm length, 1.0 - 1.5 μm width and 50 - 100 nm height. In Fig. 2(b-2, c-2 and d-2), the presences of the corresponding three crystal phases of BiPO_4 can be clearly found, respectively. On their surfaces a mass of much smaller spherical particles with size of 50 - 100 nm were dispersed randomly, which should be $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ nanoparticles. Therefore, it can be inferred that three photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$) have been synthesized. In Fig. 2(e), as a composite of three crystal phases of BiPO_4 , HBIP, nMBIP and mMBIP all can be discovered via the observation of shape and size. Also, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ nanoparticles have been well coated on their surfaces.

Fig. 2.

The EDX spectra of synthesized $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ particles and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ nanocomposite particles are all shown in Fig. 3 to further investigate the chemical compositions and states. In Fig. 3(a), the result displays the main peaks of Er, Y, Al and O elements, which demonstrates the composition of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ as up-conversion luminescence agent. Besides, the atomic ratio is almost consistent with the stoichiometric ratio of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$. From Fig. 3(b, c and d), the peaks of Bi and P elements can be clearly observed, which belong to the BiPO_4 nanoparticles, as well as all peaks of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ can also be seen easily. The corresponding ratio of each ingredient is in accord

with the theoretical calculating values.

Fig. 3.

Diffuse reflectance spectra of these three crystal phases of BiPO_4 (HBIP, nMBIP and mMBIP) are shown in Fig. 4. According to the Fig. 4(a-1, b-1 and c-1), it can be found that the absorption edges of HBIP, nMBIP, and mMBIP appear at 370 nm, 320 nm and 290 nm, respectively. As shown in the corresponding Fig. 4(a-2, b-2 and c-2), the band-gap energies are estimated to be 4.62 eV, 3.83 eV and 4.21 eV for HBIP, nMBIP and mMBIP, respectively [36]. The differences may be due to their electronic structures based on the crystal phases.

Fig. 4.

Diffuse reflectance spectra of prepared $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ were measured in the range of wavelength from 200 to 800 nm. It is displayed in Fig. 5(a) that the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ has absorption in visible-light and infrared-light regions, which indicates that, as up-conversion luminescence agent, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ can utilize most visible-light and infrared-light of solar spectrum and lay the ground for converting both them into high-energy ultraviolet-lights. According to the Fig. 5(b, c, d and e), the four photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$) still demonstrated the absorption in visible-light and infrared-light regions due to the presence of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$. Although the absorption values are relatively low due to the small proportion of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, it plays an important role to conduct the photocatalytic conversion of nitrite since the photocatalysts can continuously utilize the visible-lights and infrared-lights of solar spectrum.

Fig. 5.

The FT-IR spectra of prepared $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ are shown in Fig. 6. From Fig. 6(a) it can be confirmed that the absorption band centered around 606 cm^{-1} is resulting from anti-symmetry bending vibration of the O-P-O bond [37]. The absorption band located around 1067 cm^{-1} is resulting from the ν_3 anti-symmetry stretching vibration of the P-O bond [38]. These absorption bands are characteristic vibrations of the phosphate groups in structure of hexagon BiPO_4 (space group: P3_121 , HBIP). The absorption bands centered around 1631 cm^{-1} and 3432 cm^{-1} are probably attributed to $\delta(\text{H-O-H})$ and $\nu(\text{O-H})$ of the coordinated water to the bismuth atoms, respectively, in the BiPO_4 . Similarly, in Fig. 6(b) it can be found that the absorption bands centered around 548 cm^{-1} and 670 cm^{-1} are resulting from anti-symmetry bending vibration of the O-P-O bond. The absorption bands centered around 1010 cm^{-1} and 1077 cm^{-1} are resulting from the ν_3 anti-symmetry stretching vibration of the P-O bond. And the absorption bands centered around 1633 cm^{-1} and 3433 cm^{-1} come from $\delta(\text{H-O-H})$ and $\nu(\text{O-H})$ of the coordinated water, respectively, to the bismuth atoms in the monoclinic BiPO_4 (space group: P21/n , nMBIP). As shown in Fig. 6(c), the absorption bands among $595 - 690\text{ cm}^{-1}$ are resulting from anti-symmetry bending vibration of the O-P-O bond. The absorption bands located around 1003 cm^{-1} and 1072 cm^{-1} are resulting from the ν_3 anti-symmetry stretching vibration of the P-O bond. The absorption bands centered around 1620 cm^{-1} and 3450 cm^{-1} probably attribute to $\delta(\text{H-O-H})$ and $\nu(\text{O-H})$ of the coordinated water, respectively, to the bismuth atoms in the monoclinic BiPO_4 (space group: P21/m , mMBIP). Fig. 6(d) shows the similar results as described above including the characteristic vibrations of the phosphate groups and the $\delta(\text{H-O-H})$ and $\nu(\text{O-H})$ of the coordinated water to the bismuth atoms in three crystal BiPO_4 composites.

Fig. 6.

3.2. The influence of different photosources (ultraviolet-light, visible-light and simulated solar-light) on photocatalytic conversion conversion rate of nitrite in aqueous solution

Fig. 7 shows the influence of different photosources (ultraviolet-, visible- and simulated solar-lights) on the photocatalytic conversions of nitrite over these four photocatalysts. In addition, for three bare BiPO_4 without $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ the photocatalytic conversions of nitrite were also investigated for comparison. At first, under ultraviolet-light irradiation, for all used photocatalysts including bare BiPO_4 they displayed relatively high photocatalytic activities in the conversions of nitrite. Among them, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$ gives 86.46 % photocatalytic conversion rate of nitrite, displaying the highest photocatalytic activity. And the conversion rate of 59.80 % can also be obtained over the mMBIP. Overall, as wide-band semiconductor compounds these prepared photocatalysts with or without the up-conversion luminescence agent, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, all can display better photocatalytic activity under ultraviolet-light irradiation. Secondly, under visible-light irradiation the bismuth phosphate (BiPO_4) photocatalysts combined with up-conversion luminescence agent, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, still displayed high photocatalytic activities in the conversions of nitrite. Among them, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ shows the highest photocatalytic activity, reaching 74.85 % conversion rate of nitrite. Not surprisingly, due to the presence of up-conversion luminescence agent, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, the irradiated visible-lights can be converted into ultraviolet-light, which can effectively stimulate the wide-band bismuth phosphate (BiPO_4) compounds as photocatalysts, also displaying high photocatalytic activities in the conversions of nitrite. However, for three bare bismuth phosphates

(BiPO₄) they all revealed very low photocatalytic activity. Apparently, due to their characters as wide-band semiconductors they can be activated only under ultraviolet-light excitation.

Moreover, under simulated solar-light irradiation, like ultraviolet-light and visible-light irradiation the bismuth phosphate (BiPO₄) photocatalysts combined with up-conversion luminescence agent, Er³⁺:Y₃Al₅O₁₂, also displayed high photocatalytic activities in the conversions of nitrite. It further demonstrates that the up-conversion luminescence agent, Er³⁺:Y₃Al₅O₁₂, plays an important role in the effective photocatalytic conversion of nitrite. Although the three bare bismuth phosphates (BiPO₄) without up-conversion luminescence agent, Er³⁺:Y₃Al₅O₁₂, displayed a certain photocatalytic activity under simulated solar-light irradiation due to the presence of a small amount of ultraviolet-lights in simulated solar-lights, they are far more low than those of four photocatalysts combined with the up-conversion luminescence, Er³⁺:Y₃Al₅O₁₂. It is indicated that the simulated solar-lights, as a continuous spectrum light source, becomes ultraviolet-lights with various wavelengths through the up-conversion luminescence of Er³⁺:Y₃Al₅O₁₂. The generated ultraviolet-lights with different wavelengths can excite the mMBIP, nMBIP and HBIP with different band-gaps, respectively, to effectively perform their photocatalytic reactions. Due to the different band-gaps, the mMBIP, nMBIP and HBIP absorb 370 nm, 320 nm and 290 nm wavelengths of ultraviolet-lights from up-conversion luminescence range of Er³⁺:Y₃Al₅O₁₂, respectively. It can be found that the obtained photocatalytic conversion rates of nitrite could reached 78.62 %, 70.05 % and 82.56 %, respectively, in the presence of Er³⁺:Y₃Al₅O₁₂/(H-nM-mM)BIP under ultraviolet-, visible- and simulated solar-lights. Potentially, the Er³⁺:Y₃Al₅O₁₂/(H-nM-mM)BIP would display much higher photocatalytic activity if the molar ratio of mMBIP, nMBIP and HBIP in

$\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ were appropriately adjusted according to the component of ultraviolet-lights from $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$.

Fig. 7.

3.3. The influences of simulated solar-light irradiation time and corresponding reaction kinetics on photocatalytic conversion rate of nitrite in aqueous solution

The photocatalytic conversion of nitrite caused by four prepared photocatalysts and three bare BiPO_4 were conducted under simulated solar-light irradiation. Before light irradiation, the solutions were magnetically and constantly stirred in the dark for 30 min to reach the adsorption-desorption equilibrium. Generally speaking, due to the special surface of catalyst, the properly adsorption is helpful to the photocatalytic conversion of contaminant in aqueous solution [39,40]. Therefore, it is useful for the photocatalytic conversion of nitrite that the photocatalysts adsorb a certain amount of nitrite. Then, the solution samples were taken out every 2.0 h for examining the concentration rates of nitrite by liquid chromatograph. From Fig. 8, it can be seen that, along with increasing simulated solar-light irradiation time, the photocatalytic conversion rates of nitrite over three crystal phases of BiPO_4 (HBIP, nMBIP and mMBIP) increase very slowly, and finally reach a very low level. This is because these bismuth phosphate (BiPO_4) could be stimulated only under ultraviolet-light excitation, which accounts for less than 5.0 % of the entire solar spectrum, due to their wide band-gaps. For their corresponding photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$) and their nanocomposite photocatalyst ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$), the photocatalytic conversion rates of nitrite all increase steadily with continuous irradiation time. At beginning, along with the increase of the irradiation time, for four photocatalysts the photocatalytic conversion rates of nitrite

showed some differences. Within 4.0 h simulated solar-light irradiation, the photocatalytic conversion rate of nitrite over $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$ raised rapidly and was far higher than those by using other three photocatalysts. And then, the photocatalytic conversion rates of nitrite over these four photocatalysts raised slightly slowly. After 8.0 simulated solar-light irradiation, the photocatalytic conversion rates reached 85.36 %, 84.44 %, 82.52 % and 78.57 %, respectively, for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$. As a consequence, it is deduced that the strategies of adding up-conversion luminescence agent ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$) to acquire high photocatalytic activity under simulated solar light irradiation can be applied in the photocatalytic conversion reaction of nitrite by using wide-band semiconductor BiPO_4 .

For further comparing the photocatalytic conversion rates of nitrite caused by these photocatalysts, their first-order reaction kinetics were investigated as shown in Fig. 8. It can be seen that all $-\ln(C_t/C_0)$ data and irradiation time (t) are able to be considered as approximate linear relationships. The reaction kinetic equations are found to be $-\ln(C_t/C_0) = 0.2079t - 0.1492$ ($R^2 = 0.9083$) for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$, $-\ln(C_t/C_0) = 0.2444t - 0.1292$ ($R^2 = 0.9818$) for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$, $-\ln(C_t/C_0) = 0.2356t - 0.1865$ ($R^2 = 0.9546$) for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $-\ln(C_t/C_0) = 0.1861t + 0.0476$ ($R^2 = 0.9896$) for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $-\ln(C_t/C_0) = 0.0089t - 0.0001$ ($R^2 = 0.9998$) for mMBIP, $-\ln(C_t/C_0) = 0.0082t + 0.001$ ($R^2 = 0.9859$) for nMBIP and $-\ln(C_t/C_0) = 0.0072t + 0.0009$ ($R^2 = 0.9717$) for HBIP, respectively. The corresponding rate constants are 0.2079 min^{-1} , 0.2444 min^{-1} , 0.2356 min^{-1} , 0.1861 min^{-1} , 0.0089 min^{-1} , 0.0082 min^{-1} and 0.0072 min^{-1} , respectively, for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, mMBIP, nMBIP and HBIP. Apparently, the order of photocatalytic conversion

rates of nitrite can be judged as $\text{HBIP} < \text{nMBIP} < \text{mMBIP} < \text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP} < \text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP} < \text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP} < \text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$.

Fig. 8.

3.4. The influences of electron and hole scavengers on photocatalytic conversion of nitrite in aqueous solution

In this section, Ag^+ and EDTA were used as electron and hole scavengers, respectively, to investigate the process and mechanism of the photocatalytic conversion of nitrite caused by bismuth phosphate (BiPO_4) under simulated solar-light irradiation. As shown in Fig. 9, when the electron and hole scavengers are not used, the photocatalytic conversion rates of nitrite can reach 85.36 %, 84.44 %, 82.52 % and 78.57 %, respectively, for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$. However, after adding Ag^+ and EDTA, the photocatalytic conversion rates of nitrite all display an apparent decline for these catalytic systems. This demonstrates that Ag^+ and EDTA, as electron and hole scavengers, respectively, can compete with nitrite ions on the conduction band (CB) and valence band (VB) of semiconductor catalysts. They were effectively oxidized and reduced, respectively, which causes a decreased photocatalytic conversion rate of nitrite. It indicates that the oxidation and reduction reactions of the NO_2^- ions should occur on the conduction band (CB) and valence band (VB), respectively, producing the NH_4^+ , N_2 and NO_3^- .

Fig. 9.

3.5. The influences of used times on photocatalytic conversion rate of nitrite in aqueous solution over $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ photocatalyst

It is worth pointing out that the stability of the photocatalysts is a crucial factor for their practical applications. In order to examine the stability and repeatability of the prepared four photocatalysts, three recycling tests were conducted under simulated solar light irradiation as shown in Fig. 10. It can be found that, for four photocatalysts, the photocatalytic conversion rates of nitrite all decrease slightly along with the increase of used times, which indicates that the photocatalytic activities of these four prepared photocatalysts decrease only slightly after each (8.0 h) use. Particularly, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ displays the least decrease in the photocatalytic activity for four photocatalysts. That is to say, there is no significant decrease for photocatalytic conversion rate of nitrite caused by $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ after total 24 h irradiation time. It reveals that the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ can be used for long time to highly effectively convert nitrite under simulated solar light irradiation.

Fig. 10.

3.6. Possible mechanism on simulated solar-light photocatalytic conversion of nitrite in the presence of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ photocatalysts

The possible mechanism on photocatalytic conversion of nitrite for the prepared photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$) under simulated solar-light is proposed in Fig. 11. $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$, as an up-conversion luminescence agent, deposited on the surface of BiPO_4 particles, can convert visible-light in simulated solar-light into ultraviolet-light, which can effectively excite the wide-band gap BiPO_4 , leading electrons to transfer from the valence band (VB) to conduction band (CB) generating electron (e^-) - hole (h^+) pairs. In previous studies, for $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ excited by 652.2 nm the emission bands 354 - 359 nm converting could be detected [41].

Otherwise, 318.7 nm and 320.1 nm can be observed under the converting of 486.5 nm and 542.4 nm (or 548.8 nm), respectively [42,43]. In addition, when the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ is pumped by a 488 nm Ar^+ laser, the up-conversion signal at 271 nm can be observed [44]. The up-conversion fluorescence spectrum and energy-level diagram showing related transitions were given in corresponding literatures [45,46].

According to the Fig. 4, the absorption edges of HBIP, nMBIP and mMBIP, which occur at about 370 nm, 320 nm and 290 nm, matched well with the emission bands of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ effectually in above three conditions. Under multiple-pulsed excitation, the photon avalanche may take place. The simulated solar light section contains continuous pump wavelengths used as an excitation source that can excite $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ to give discontinuous emissions of ultraviolet-lights. Therefore, HBIP, nMBIP and mMBIP are more effectively activated to generate the electron (e^-) - hole (h^+) pairs. These generated electrons (e^-) quickly take part in reduction reaction, so most of NO_2^- ions in aqueous solution are easily reduced to NH_4^+ ($\text{pH} \leq 7.0$) or N_2 ($\text{pH} \geq 7.0$). Meanwhile, the generated holes (h^+) on valence band (VB) are consumed for converting NO_2^- to NO_3^- . As a result, the corresponding photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$) are proved to have high photocatalytic activity. And their nanocomposite photocatalysts ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$) also exhibit superior photocatalytic performance. Perhaps, the photocatalytic activity of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ can be further enhanced if the molar ratio of HBIP, nMBIP and mMBIP is adjusted based on the component of ultraviolet-light resulted from $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$.

The possible mechanism for the simulated solar-light photocatalytic conversion of nitrite over $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{BiPO}_4$ nanocomposite was as follows:

Fig. 11.

4. Conclusions

In this study, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ for solar light photocatalytic conversion of nitrite in aqueous solution were successfully prepared by sol-gel, hydrothermal, ultrasonic dispersion and liquid boiling methods. Due to the use of up-conversion luminescence agent ($\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$), they all exhibited excellent photocatalytic activity under simulated solar light, compared with that under visible-light and ultraviolet-light. Particularly, since HBIP, nMBIP and mMBIP have different band gaps, as a such composite the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ plays a much more photocatalytic activity, resulting in 82.56 % under simulated solar light irradiation. Therefore, the $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ as a wide spectrum semiconductor photocatalyst is suitable to be applied in dealing with nitrite by utilizing solar energy. In addition, these prepared photocatalysts can be effectively recycled for three repetitive cycles without an apparent deactivation on the photocatalytic activity. It is wished that these research results could provide a possible strategy for utilization of solar energy and purification of drinking water in the future.

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Content of Figures:

Fig. 1. XRD patterns of (a) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ (heat-treated at 1100 °C for 2.0 h), (b-1) HBIP (at 20 °C for 72 h), (c-1) nMBIP (at 100 °C for 72 h), (d-1) mMBIP (at 200 °C for 72 h), (b-2) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and HBIP mass ratio), (c-2) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and nMBIP mass ratio), (d-2) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and mMBIP mass ratio) and (e) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and (H-nM-mM)BIP mass ratio).

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Fig. 3. EDX patterns of (a) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ (heat-treated at 1100 °C for 2.0 h), (b) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and HBIP mass ratio), (c) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and nMBIP mass ratio) and (d) $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and mMBIP mass ratio).

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Fig. 7. The influences of different photosources (ultraviolet-light, visible-light and simulated solar-light) on

photocatalytic activities of HBIP (at 20 °C for 72 h), nMBIP (at 100 °C for 72 h), mMBIP (at 200 °C for 72 h), $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and HBIP mass ratio), $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and nMBIP mass ratio), $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and mMBIP mass ratio) and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/(\text{H-nM-mM})\text{BIP}$ (0.3:1.0 $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ and (H-nM-mM)BIP mass ratio).

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Fig. 11. Possible mechanism on simulated solar-light photocatalytic conversion of nitrite in the presence of $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{HBIP}$, $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{nMBIP}$ and $\text{Er}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}/\text{mMBIP}$.

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