



PbCrO₄ yellow-pigment nanorods: An efficient and stable visible-light-active photocatalyst for O₂ evolution and photodegradation

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ABSTRACT Here, PbCrO₄ nanorods, a commonly used and low-cost yellow pigment, was synthesized *via* a simple precipitation reaction and can serve as a highly efficient oxygen production and photodegradation photocatalyst. The obtained PbCrO₄ nanorods exhibit excellent stability and photocatalytic performance for O₂ evolution from water. The production rate is approximately 314.0 μmol h⁻¹ g⁻¹ under visible light, and the quantum efficiency is approximately 2.16% at 420±10 nm and 0.05% at 600±10 nm. In addition, the PbCrO₄ shows good degradation performance for methylene blue, methyl blue, methyl orange and phenol under visible-light irradiation. These results indicate that it is potential to fabricate an effective, robust PbCrO₄ photocatalyst by transforming heavy-metal pollutants Pb(II) and Cr(VI) into a highly efficient O₂ evolution and photodegradation material. This strategy which uses pollutant to produce clean energy and degrade contaminants is completely green and environmentally benign, and thus could be a promising way for practical environmental applications.

Keywords: O₂ evolution, pollutant, PbCrO₄ nanorods, visible-light-active, photocatalyst

INTRODUCTION

Photocatalytic water splitting is one of the most promising approaches to address energy and environmental issues [1–3]. Water splitting can be separated into two half reactions, namely, hydrogen evolution and oxygen evolution.

The generation of H₂ by a photocatalyst has been heavily researched over the past several years, and the generation rates have been significantly improved. However, the O₂ generation reaction (water oxidation) is still difficult due to the multiple step reactions that involve four electrons with low reaction rate of about 5 orders of magnitude slower than that of the H₂ evolution. Therefore, O₂ evolution remains the major obstacle for the overall water-splitting reaction [4–6]. The basic requirement in O₂ generation is that the valence band of the photocatalyst must be more positive than the oxidation potential of H₂O to O₂ (1.23 V *versus* normal hydrogen electrode (NHE), pH=0). Furthermore, a significant overpotential is required to excite four electrons. Although a variety of materials have been developed over the past few decades for this purpose, very few materials reported can directly oxidize water into O₂ under visible-light illumination [7].

Monoclinic bismuth vanadate (BiVO₄), which has a relatively narrow bandgap of ~2.4 eV and highly positive valence band edge of 2.86 V *versus* NHE, is a low-cost, photostable photocatalyst that has been widely used in solar energy conversion. However, the low mobility and the high recombination rates of the photogenerated charge carriers in BiVO₄ usually result in a poor photocatalytic activity, which has seriously hampered its practical applications, especially for photocatalytic O₂ evolution [8,9]. Recently, silver orthophosphate (Ag₃PO₄)

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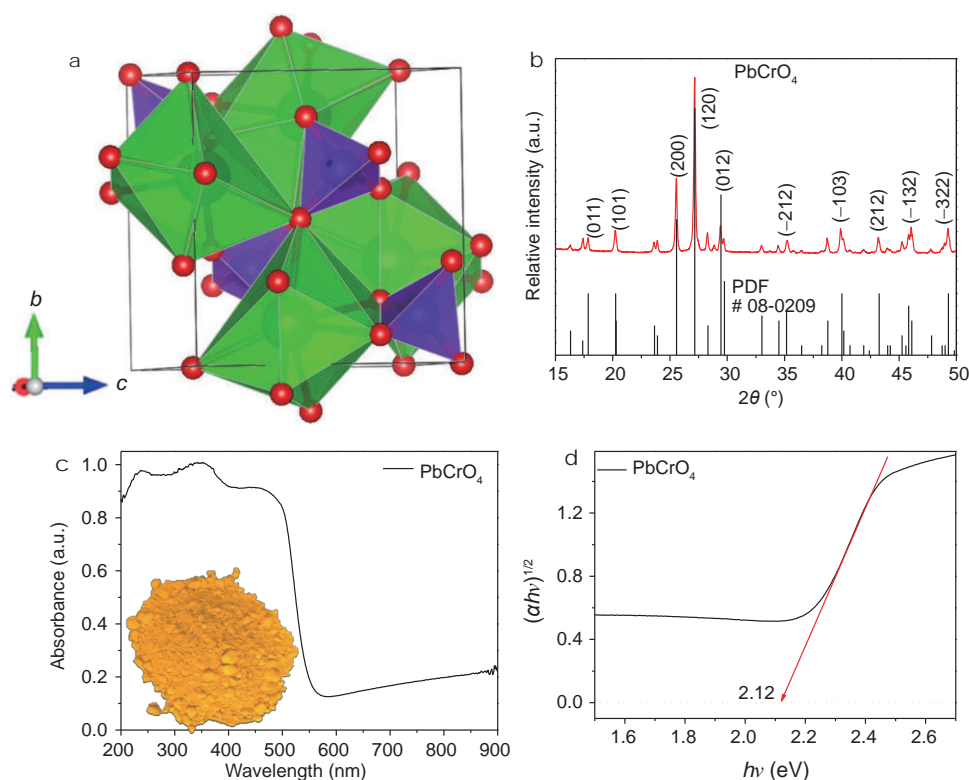


Figure 1 The crystal structure (Pb, green; Cr, purple; O, red) of PbCrO₄ (a). The XRD pattern (b), DRS UV-vis spectrum (c), optical image (inset of c) and Tauc plot of the transformed Kubelka-Munk function *versus* the energy (d) of the PbCrO₄ nanorods.

has been reported to be one of the few materials that exhibit an excellent visible-light-driven photooxidative capability for O₂ evolution from water, but Ag₃PO₄ is unstable under light irradiation and suffers from photocorrosion as a result of its self-reduction to silver by the photogenerated electrons [7,10,11]. In addition, the noble metallic composition may limit practical applications. Other O₂ evolution photocatalysts, such as TaON [12], Ta₃N₅ [13], Fe₂O₃ [14] and WO₃ [15], either are unstable under illumination or have weak photocatalytic performances. Developing highly efficient, low-cost and stable visible-light-active photocatalysts for O₂ evolution from water is a challenge.

Lead chromate (PbCrO₄), one of the most commonly used inorganic yellow pigments in plastics, coatings and paints, could be a good visible-light-active photocatalyst due to its excellent photostability, thermostability, easy preparation and low cost [16–18]. Herein, using a simple precipitation reaction, we transformed heavy-metal Pb(II) and Cr(VI) pollutants into the yellow pigment, PbCrO₄, as a stable and efficient visible-light-active photocatalyst for O₂ evolution from water with negligible pollutant residues during the photocatalytic reaction. In addition,

PbCrO₄ has a good decomposition performance for methylene blue, methyl blue, methyl orange and phenol under visible-light irradiation. This novel and efficient photocatalyst may result in breakthroughs for the overall water-splitting reaction and specially for the reuse of pollutants containing Pb(II) and Cr(VI).

RESULTS AND DISCUSSION

The PbCrO₄ nanorods were synthesized by a precipitation reaction of Pb(NO₃)₂ and Na₂CrO₄ solutions (see Supplementary information for detail). The crystal structure and unit cell of PbCrO₄ ($a = 7.12$ Å, $b = 7.44$ Å, $c = 6.8$ Å; $\alpha = 90.0^\circ$, $\beta = 102.4^\circ$, $\gamma = 90.0^\circ$) are shown in Fig. 1a. The corner-connected PbO₉ twisted enneahedron and CrO₄ tetrahedron are alternately arranged in the crystal structure. The X-ray diffraction pattern (XRD) of the as-synthesized PbCrO₄ (Fig. 1b) exhibits a typical monoclinic phase with diffraction peaks at 17.87°, 20.31°, 25.58°, 27.17°, 29.46°, 35.18°, 40.00°, 43.25°, 45.84° and 49.30°, which are related to the (011), (101), (200), (120), (012), (-212), (-103), (212), (-132) and (-322) facets of PbCrO₄ (JCPDS No. 08-0209). The energy dispersive X-ray analysis (EDX) spectrum (Fig. S1) shows the molar ratio of

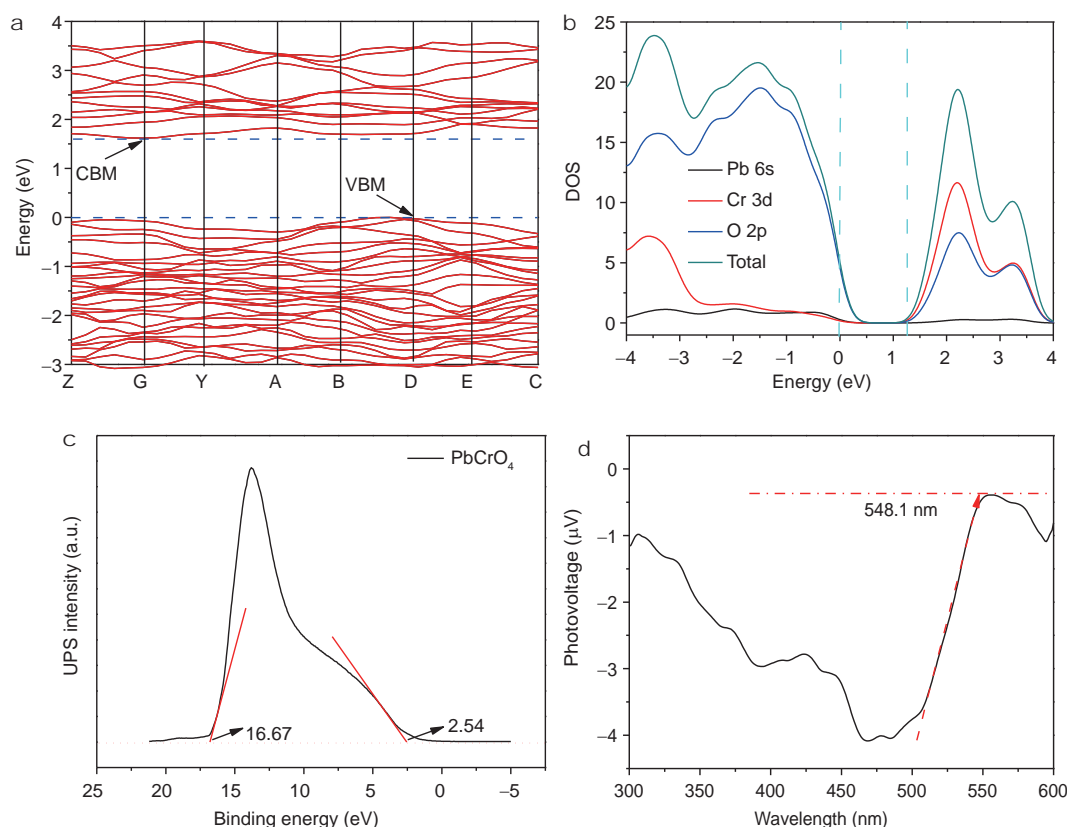


Figure 2 The calculated electronic band structures (a), DOS (b), UPS (c) and SPS (d) of the PbCrO_4 nanorods.

Pb, Cr and O is close to 1:1:4, indicating stoichiometric PbCrO_4 was successfully prepared. Diffuse reflectance UV-vis spectroscopy (DRS) was used to evaluate the absorption region of PbCrO_4 , as presented in Fig. 1c. The deep yellow powders (Inset of Fig. 1c) have a strong absorption band in the visible-light region. The band gap of PbCrO_4 can be calculated to be 2.12 eV from the Tauc plot (Fig. 1d).

To better understand the electronic structure of PbCrO_4 , we conducted density functional theory (DFT) calculations. Fig. 2a shows the calculated band structure of PbCrO_4 with an indirect band gap of 1.62 eV at the D and G point, which is 0.50 eV smaller than the experimental value (2.12 eV) due to the well-known standard DFT band gap underestimation. The density of state (DOS) calculation (Fig. 2b) indicates that the valence band maximum (VBM) of PbCrO_4 originates from the O 2p state, and the conduction band minimum (CBM) mainly originates from Cr 3d. The Pb 6s state has a relatively less contribution to the conduction band. Ultra-violet photoelectron spectroscopy (UPS) was used to determine the valence band energy (E_{VB}) of PbCrO_4 (Fig.

2c), which is 2.65 V *versus* the reversible hydrogen electrode (RHE) and matches the calculated E_{VB} (2.57 V *versus* the RHE) from the empirical equation (Table S1). Because the E_{VB} value is 1.42 and 0.95 V higher than the potentials of $\text{O}_2/\text{H}_2\text{O}$ (1.23 V) and $\text{OH}^\cdot/\text{OH}$ (1.70 V), PbCrO_4 may have photocatalytic activity for O_2 evolution from water splitting and $\cdot\text{OH}$ radical generation. The conduction band energy (E_{CB}) is approximately 0.53 V *versus* the RHE based on $E_{\text{VB}} - E_{\text{g}}$. Surface photovoltage spectroscopy (SPS) is a powerful tool to characterize the charge separation and the photoelectric response on the nanoscale [19]. As shown in Fig. 2d, SPS shows an active negative surface photovoltage (SPV) response, indicating PbCrO_4 is a p-type semiconductor [19]. The SPV response reaches 548.1 nm, which agrees with the solid PbCrO_4 powder absorption (Fig. 1c). Furthermore, the strong SPV response illustrates that the generated carriers can efficiently separate and rapidly migrate to the surface, indicating PbCrO_4 may have a good photocatalytic performance.

The field emission scanning electron microscopy (FE-SEM) images (Fig. 3a, b) and low-resolution transmission

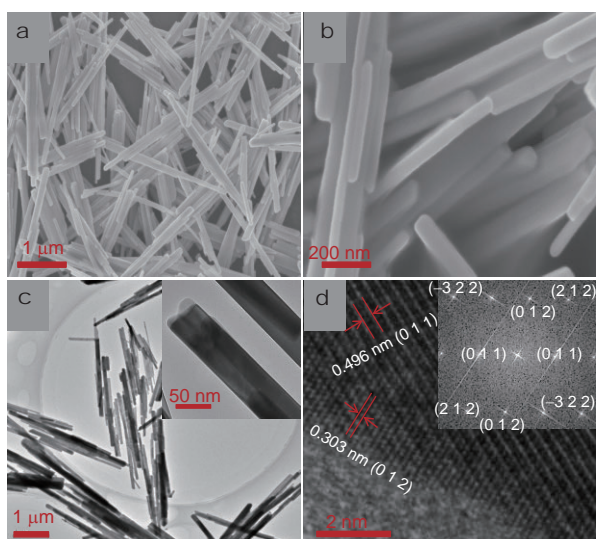


Figure 3 The FE-SEM images (a, b), low-resolution TEM image (c) and HR-TEM image (d) of the PbCrO_4 nanorods.

electron microscopy (TEM) images (Fig. 3c and inset) show the PbCrO_4 is single-crystal nanorods with 1–5 μm in length and 50–100 nm in diameter. The characteristic lattice fringe spacing of monoclinic phase PbCrO_4 , (011) and (012), is 0.496 and 0.303 nm, respectively, observed from the high resolution TEM (HR-TEM) images (Fig. 3d). In order to interpret the synthetic mechanism of nanorod-like PbCrO_4 , we further characterized the morphology of PbCrO_4 with SEM, as the reaction time prolonged. As shown in Fig. S2, the PbCrO_4 prepared at 180 min was obviously longer than that of 10 min. Accordingly, the nanorod-like PbCrO_4 is grown through three processes: the formation of PbCrO_4 crystal nuclei, short nanorod (Fig. S2a) and further growth to the long nanorod (Fig. S2b), where the surface energy could be the

mainly driving force for the formation of nanorod. The Brunauer-Emmett-Teller (BET) surface area and pore size of the PbCrO_4 nanorods are characterized using a N_2 adsorption-desorption isotherm, as shown in Fig. S3. The isotherm is type-III with an H4-type hysteresis loop, indicating a macroporous structure mainly caused by the stacking of the PbCrO_4 nanorods [20–22]. The BET surface area is $8.50 \text{ m}^2 \text{ g}^{-1}$. The high-resolution XPS spectra of Pb 4f and Cr 2p are shown in Fig. S4. The binding energies at 142.9, 138.2, 588.1 and 578.9 eV are assigned to Pb 4f_{5/2}, Pb 4f_{7/2}, Cr 2p_{1/2} and Cr 2p_{3/2}, respectively, which is in agreement with the reported binding energy values of PbCrO_4 [23,24].

The above results (DRS, SPS and UPS) show that the PbCrO_4 nanorods have good visible-light harvesting, photoexcited charge separation, and suitable energy band positions, and they can be used for O_2 evolution by water splitting under visible light. Commonly used electron sacrificial agents, such as AgNO_3 , FeCl_3 , $\text{Na}_2\text{S}_2\text{O}_8$ are adopted to evaluate the photocatalytic activity for oxygen production over PbCrO_4 nanorods. As shown in Fig. S5, the PbCrO_4 nanorods exhibits the highest oxygen production with the rate of $15.70 \mu\text{mol h}^{-1}$ ($\lambda > 420 \text{ nm}$) under AgNO_3 (0.1 mol L^{-1}) as electron sacrificial agents. Fig. 4a shows the O_2 evolution in the PbCrO_4 nanorods and BiVO_4 synthesized by a liquid-solid reaction according to Kudo's report [8] under visible-light irradiation and the AgNO_3 (0.1 mol L^{-1}). The O_2 evolution rate of PbCrO_4 is higher than that of the as-synthesized BiVO_4 . A comparison of the normalized O_2 evolution rates with many other reported visible-light-active photocatalysts is shown in Table S2. Clearly, the photocatalytic performance of the synthesized PbCrO_4 nanorods is at the top among many reported visible-light-active photocatalysts (polymer carbon nitride, PDINH

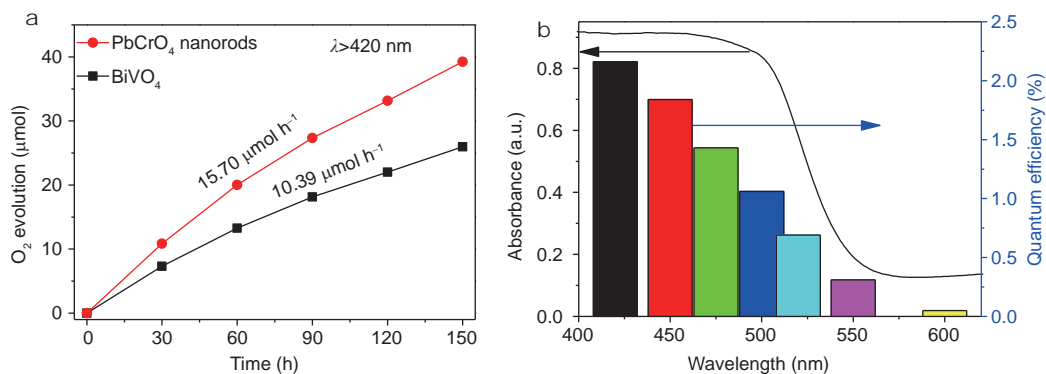


Figure 4 The O_2 evolution of the PbCrO_4 nanorods and BiVO_4 under visible-light irradiation (a), the measured quantum efficiencies for photons at different wavelengths for the PbCrO_4 nanorods (b).

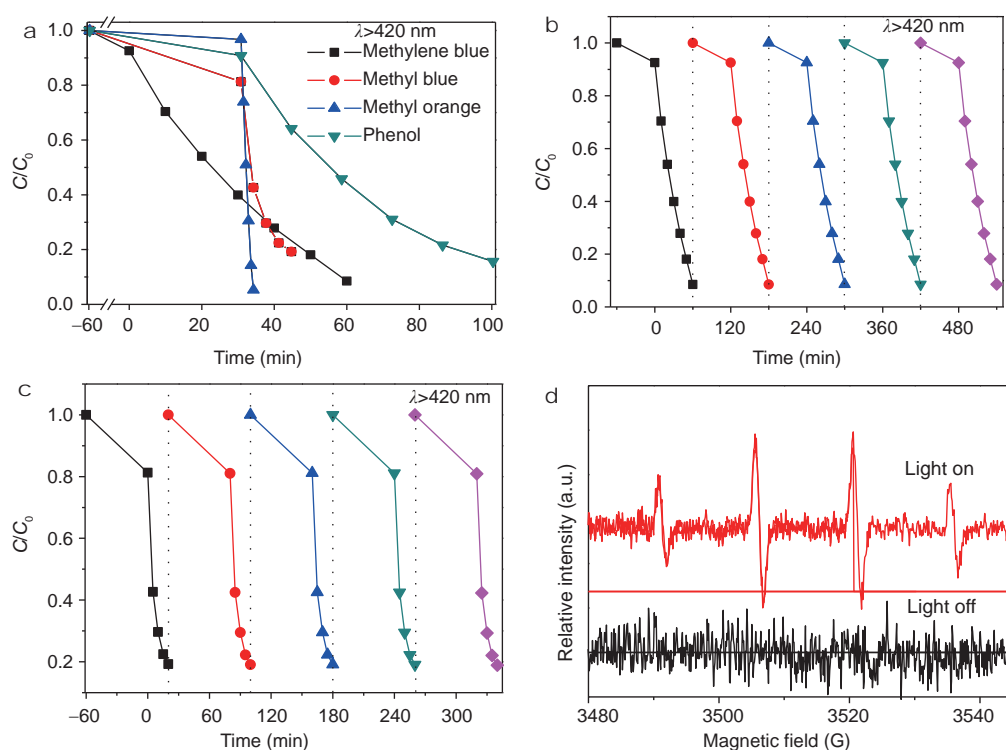


Figure 5 The photocatalytic degradation ($\lambda > 420$ nm) of dyes (a), recycles measure of photocatalytic degradation of methylene blue (b) and methyl blue (c), the DMPO spin-trapping ESR spectra of PbCrO₄ nanorods for $\cdot\text{DMPO}\cdot\text{OH}$ (d).

supramolecular, red phosphorus, TaON, Ta₃N₅, Sm₂Ti₂S₂O₅, Sr_{0.9}NbO₃, Fe₂O₃, AgPO₄ and BiVO₄). Fig. 4b displays the measured quantum efficiencies for photons at different wavelength for the O₂ evolution reaction over PbCrO₄ nanorods. The quantum efficiency is 2.16% and 0.05% at 420 \pm 10 and 600 \pm 10 nm, respectively.

The photocatalytic decomposition of organics was used to further evaluate the photocatalytic performance of the PbCrO₄ nanorods. As shown in Fig. 5a and Figs S7–S10, the PbCrO₄ nanorods have excellent photocatalytic degradation performances for methylene blue, methyl blue, methyl orange and phenol. What's more, after five decomposition cycles for methylene blue and methyl blue (Fig. 5b, c), no recession in the photocatalytic activity is observed, indicating the excellent stability of the PbCrO₄ nanorods. To clarify the generation of $\cdot\text{OH}$ radicals during the photodegradation reaction, the electron spin resonance (ESR) tests were performed using DMPO spin-trapping, as shown in Fig. 5d. Four characteristic peaks are observed under visible-light irradiation, and the standard ratios of the intensities are 1:2:2:1, which can be ascribed to $\cdot\text{DMPO}\cdot\text{OH}$ [25,26]. In contrast, an ESR signal is not detected under dark conditions, indicating that the $\cdot\text{OH}$ radical is the active species during the

photocatalytic decomposition. We supplemented the active species trapping experiments to further verify the possible active species in photocatalytic decomposition. The isopropyl alcohol (IPA), ammonium oxalate (AO), and benzoquinone (BQ) were used as scavengers of $\cdot\text{OH}$, H⁺, and $\cdot\text{O}_2^-$, respectively in the photocatalytic decomposition of methyl blue [27–29]. As shown in Fig. S11, the photodegradation rates are apparently inhibited by the addition of IPA and AO, while BQ exhibits a relatively weak inhibition. These results suggest that $\cdot\text{OH}$ and H⁺ are the main active species in the photocatalytic decomposition.

We next investigated the pollution from PbCrO₄ by examining the concentrations of Pb²⁺ and Cr⁶⁺ that dissolved into the water. The solubility product (K_{sp}) of PbCrO₄ is 2.8×10^{-13} , and the concentrations of Pb²⁺ and Cr⁶⁺ are 0.110 and 0.028 ppm, respectively, which are close to the drinking water standard concentrations for Pb²⁺ (0.05 ppm) and Cr⁶⁺ (0.05 ppm) and lower than the industrial sewage emission standards for Pb²⁺ (1.00 ppm) and Cr⁶⁺ (1.00 ppm). The inductively coupled plasma optical emission spectrometry (ICP-OES) tests (Table S3) further clarify that Pb and Cr element remain in the reaction solution after the photocatalytic recycling, and the

concentrations are only 0.16 ± 0.02 and 0.00 ppm, respectively. These results show that we can transform heavy-metal Pb(II) and Cr(VI) pollutants into a visible-light-active photocatalyst and produce negligible pollutant residues during the photocatalytic reaction.

CONCLUSIONS

In summary, the PbCrO_4 nanorods were successfully developed by a simple precipitation reaction *via* mixing $\text{Pb}(\text{NO}_3)_2$ and Na_2CrO_4 solutions at room temperature. The obtained nanorods are visible-light-active photocatalyst for high-efficiency photocatalytic O_2 evolution from water and photocatalytic organic pollutants degradation. This simple synthetic method can transform heavy-metal Pb(II) and Cr(VI) pollutants into low toxic PbCrO_4 photocatalyst with a narrow band gap of 2.12 eV, excellent stability and high photocatalytic performance and produce negligible pollutant residues during the photocatalytic reaction. This novel photocatalyst may result in the breakthrough in the overall water-splitting reaction, degradation of pollutants and the reuse of Pb(II) and Cr(VI) pollutants.

Received 9 December 2017; accepted 9 February 2018;
published online 15 March 2018

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Acknowledgements This work was jointly supported by the National Natural Science Foundation of China (21401190), the Science and Technology Project of Research Foundation of China Postdoctoral Sci-

ence (2017M612710 and 2016M592519), Shenzhen Peacock Plan (827-000059, 827-000113 and KQTD2016053112042971), the Science and Technology Planning Project of Guangdong Province (2016B050501005), and the Educational Commission of Guangdong Province (2016KCXTD006 and 2016KSTCX126).

Author contributions Zhang GQ performed the experiments and wrote the manuscript with the guidance from Su CL and Sun X. All authors contributed to the general discussion and revision.

Conflict of interest The authors declare no conflict of interest.

Supplementary information Supporting information is available in the online version of the paper.



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铬酸铅纳米棒: 一种高效、稳定的可见光产氧和光降解催化剂

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摘要 本文利用简单的沉淀反应将工业废水中含有的剧毒Pb(II)和Cr(VI)离子制备成纳米棒状的黄色颜料PbCrO₄。合成的PbCrO₄纳米棒是一种高效的可见光活性产氧和光降解催化剂。此纳米棒具有优异的光催化稳定性和催化活性, 在可见光下分解水产氧速率高达314.0 μmol h⁻¹ g⁻¹, 在420±10 和600±10 nm处量子效率分别为2.16%和0.05%。此外, 该催化剂还具有良好的可见光降解亚甲基蓝、甲基蓝、甲基橙和苯酚的性能。以上结果表明, 将剧毒含重金属污染物Pb(II)和Cr(VI)的离子溶液转化为环境友好且高效的可见光光催化剂是可行的。该策略利用污染物产生清洁能源并降解污染物, 是绿色、环境友好且非常有前景的废物利用途径。