



Enhancement of the photocatalytic performance of Ni-loaded TiO₂ photocatalyst under sunlight

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Abstract

A highly sunlight active Ni-loaded TiO₂ nanocomposite (Ni/TiO₂) is successfully prepared by a simple chemical reduction method using tetrabutyl titanate as a precursor, Ni(NO₃)₂·6H₂O as a nickel source, and N₂H₄·H₂O as a reductant, respectively. The crystal structure, morphology and UV–vis diffuse reflectance characteristics are investigated by XRD, TEM, and UV–vis diffuse reflectance spectroscopy, while the photocatalytic performance of Ni/TiO₂ is evaluated by photocatalytic degradation of methyl orange solution under UV and sunlight irradiation. Results show that the crystal structure of TiO₂ is not changed upon the loading of Ni, the photocatalytic performance of TiO₂ under both UV-light and sunlight, however, is enhanced greatly. The enhancement of the photocatalytic performance of Ni/TiO₂ is attributed to the increase of the photogenerated electron–hole separation efficiency and the advanced absorption of light due to surface plasmon effect of Ni nanoparticles. © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: Ni loading; Ni/TiO₂ nanocomposites; Photocatalytic activity; Mechanism

1. Introduction

The existence of organic pollutants in water and wastewater is a primary cause of serious pollution problems which can influence human health, and there requires a complete removal of these toxic and carcinogenic organic dyes [1,2]. Anatase phase of TiO₂ is the most preferable photocatalyst that extensively used in degradation and mineralization of organic dyes due to its high efficiency, strong oxidizing power, non-toxicity, high photochemical stability, and low cost [3]. However, the photocatalytic efficiency of TiO₂ is hampered by the main drawback of rapid recombination of photo-generated electron–hole pairs. To avoid the limitation, the modification of TiO₂ with metal nanoparticles is an effective technology to obtain more efficient photocatalytic activity. Several research workers have reported that loading some effective noble metals, especially Au, Ag, and Pt nanoparticles, on the surface of TiO₂ can significantly enhance the photocatalytic performance of TiO₂ [4–8]. However, such precious metals are very expensive in the view point of practical application. In addition, some authors have employed effective non-precious transition metals, including Fe [9,10], Cu [11,12],

Cr [13], Mn [14], Co, W [15], and so on, to synthesize composite for the enhancement of photocatalytic activity of TiO₂.

Ni nanoparticles, especially supported Ni nanoparticles on various carriers, have been extensively studied for the catalytic activity enhancement [16–19]. Recently, Ni/TiO₂ composite has been studied. For instance, Wu et al. has prepared Ni/TiO₂ nanocomposite by electroless plating technique for selective hydrogenation of p-nitrophenol to p-aminophenol [20]. However, the synthetic method they adopted is slightly complicated. Meanwhile, the photocatalytic activity has not been studied. In order to enhance the photocatalytic performance of TiO₂ both under UV and sunlight, in this paper, Ni-loaded TiO₂ nanocomposite is prepared by a simple chemical reduction method, and the photocatalytic activity of Ni/TiO₂ has been evaluated. Results show that the photocatalytic activity of TiO₂ has been improved greatly by Ni loading under both UV-light and sunlight irradiation.

2. Experimental

2.1. Synthesis of TiO₂ nanoparticles

TiO₂ nanoparticles were synthesized by the sol–gel method [21, 22] using tetrabutyl titanate (TBT) as a precursor. The as-prepared

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TiO₂ nanoparticles were annealed at 500 °C for 2 h to convert the amorphous TiO₂ to the crystalline form.

2.2. Preparation of Ni-loaded TiO₂ (Ni/TiO₂)

The Ni-loaded TiO₂ (Ni/TiO₂) photocatalyst was prepared according to the following steps: Firstly, the as-annealed TiO₂ nanoparticles were suspended in 50 mL distilled water and sonicated for 1 h to well disperse the nanoparticles. Then N₂H₄·H₂O was charged into the suspension and sonicated for 10 min. Second, the solution containing 5 mg of AgNO₃ (notes: which is used as nucleation sites) and the required amount of Ni(NO₃)₂·6H₂O was added dropwise, in which the added amount of Ni was x , and $x=3\%$, 6%, 9% and 12%, calculated theoretically as molar ratio of loaded Ni and TiO₂ ($x=n(\text{Ni}):n(\text{TiO}_2)$). The solution was kept reaction for 2 h with stirring after the Ni solution was dripped off. During the reaction, the color of the suspension gradually turned gray, indicating Ni²⁺ has been reduction to Ni. Finally, the obtained powder was separated from the suspension by centrifugation under 1500 rpm, and washed for three times with distilled water and ethanol. Then the resulting product was dried at 60 °C for 24 h.

2.3. Photocatalytic activity investigation and evaluation

The photocatalytic activity of TiO₂ and Ni/TiO₂ was evaluated by monitoring the photodegradation of methyl orange (MO) in aqueous solution, respectively. 0.20 g of photocatalyst of TiO₂ or Ni/TiO₂ was charged into 50 mL of 10 mg/L MO aqueous solution, respectively. The mixed solution was irradiated using high-pressure mercury lamp ($\lambda_{\text{max}}=365$ nm) under stirring. Samples were then taken out every 30 min and the photocatalyst was separated from the mixture solution by centrifugation immediately, and then the UV–vis absorption of the clarified solution was analyzed with a UV–vis spectrometer (UV-2550, Shimadzu). The absorbance of MO solution was measured at a wavelength of 465 nm, which corresponds to the maximum absorption wavelength of MO.

The photocatalytic activity of TiO₂ and Ni/TiO₂ was also investigated under sunlight irradiation. 0.20 g of photocatalyst of TiO₂ or Ni/TiO₂ was charged into 50 mL of 10 mg/L MO aqueous solution, respectively. The mixed solution was irradiated using sunlight under stirring. Samples were then taken out every 5 min and the photocatalyst was separated from the mixture solution by centrifugation immediately, and then the UV–vis absorption of the clarified solution was analyzed with a UV–vis spectrometer (UV-2550, Shimadzu). The absorbance of MO solution was measured at 465 nm, which corresponds to the maximum absorption wavelength of MO.

2.4. Characterization

The morphology was observed with transmission electron microscopy (TEM, Tecnai G2F30), the X-ray diffraction

(XRD) patterns were measured using a X-ray diffractometer (XRD-7000, Shimadzu), and the UV–vis diffuse reflectance spectra were characterized with a UV–vis spectrometer (UV-2550, Shimadzu).

3. Results and discussion

3.1. Structure of Ni/TiO₂

X-ray diffraction patterns of TiO₂ and Ni/TiO₂ are presented in Fig. 1. The diffraction pattern of TiO₂ agrees well with that in JCPDS 21-1272 card for TiO₂. The diffraction peaks present at 25.4°, 37.6°, and 48.2° are typical patterns of anatase, which contribute to the (101), (004) and (200) crystal plane, respectively. It can also be seen from Fig. 1 that both pure TiO₂ and TiO₂ in Ni/TiO₂ present in anatase phase, while the loading of Ni does not influence the crystallization performance of TiO₂. In addition, the reflection peak of Ni in Ni/TiO₂ presents at $2\theta=44.6^\circ$, which is marked with an asterisk, and it accords well with that in JCPDS 04-0850 card for Ni with face centered cubic (FCC) structure. The reflection peak corresponds to the Ni (111) crystal plane, manifesting that Ni has been loaded on TiO₂ successfully.

3.2. Morphology of Ni/TiO₂

The morphology of TiO₂ and Ni/TiO₂ is displayed in Fig. 2. The morphology of TiO₂ (see Fig. 2a) indicates that TiO₂ after annealing have sheet structure and agglomerate to some degree to form particles with 20–40 nm in size. The morphology of Ni/TiO₂ ($x=6\%$) (as shown in Fig. 2b) displays that spherical Ni nanoparticles with around 10–20 nm in size are clearly dispersed on the external surface of TiO₂ nanoparticles. Because of the difference in electron density, TiO₂ and Ni nanoparticles are distinguishable and thus Ni nanoparticles can be identified as dark spots on TiO₂ nanoparticles.

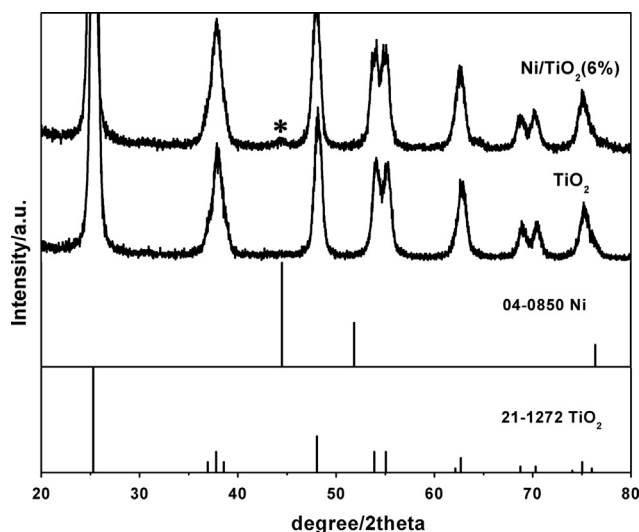


Fig. 1. XRD patterns of TiO₂ and Ni/TiO₂ ($x=6\%$).

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