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Unpredictable adsorption and visible light induced decolorization of nano rutile for the treatment of crystal violet

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ABSTRACT

Photocatalysts containing different ratios of anatase and rutile are prepared via heat treatment of Degussa P-25 titania. X-ray diffraction (XRD), Bruuauer-Emmett-Teller (BET), ultraviolet–visible light diffuse reflectance spectra (DRS), Raman spectra (Raman), positron annihilation lifetime spectra (PAL) and temperature-programmed desorption (TPD) are applied to investigate the phase composition of the synthesized catalysts. Using crystal violet (CV) as the target pollutant, the unexpected visible light decolorization of rutile is observed. Despite the decreased specific surface area, the as-synthesized rutile samples exhibit much higher adsorption capability of CV than P-25 does, which in turn leads to improved photoreaction efficiency. Since the rutile samples can't absorb the visible light, the degradation under visible light irradiation is attributed to self-sensitization of CV on the surface of rutile.

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1. Introduction

Semiconductor photocatalysis is a powerful tool for pollution control [1–3]. As an important photocatalyst, TiO_2 has received widespread concern [4–6]. The photocatalytic activity of TiO_2 is closely related to its phase composition [7]. Although rutile ($E_g = 3.0$ eV) can absorb more light than anatase ($E_g = 3.2$ eV), and it is more stable than anatase [8], it is generally agreed that anatase is a better photocatalyst. There are mainly two reasons: the first, there are more adsorbed oxygen molecules on the surface of anatase [9], and it is a crucial factor during the photoreaction; the second, anatase always has a larger specific surface area [10,11], which can provide more opportunities to contact with pollutant molecules. Therefore, there are fewer researches on rutile than that on anatase in the field of photocatalysis.

Except phase composition, the other factors, such as specific surface area, adsorption ability, and surface chemical condition, also have effects on the photocatalytic activity [12–15]. Rutile sometime can also exhibit high photocatalytic activity [16–18]. Páez et al. [16] had synthesized H_2-reduced TiO_2 by calcinations under H_2 flow. It was found that rutile with low specific surface area obtained high adsorption ability and visible photoactivity. This unusual phenomenon indicates that rutile may have potential in dealing with some kinds of dyes under visible light irradiation. However, in order to make deeper research, it is necessary to evaluate the adsorption abilities and visible photoactivity of TiO_2 prepared without H_2 treatment.

In this paper, adsorption abilities and decolorization efficiency of TiO_2 containing different ratios of anatase/rutile are investigated. Different from the usual results, rutile prepared by simple heat treatment of Degussa P25 obtains the highest visible decolorization efficiency in degrading crystal violet (CV). Characterization techniques are used to study the phenomenon, and the possible mechanism is also discussed.

2. Experiment

2.1. Materials

Titania P25 was product of Degussa Co. Ltd. CV was purchased from Shanghai Yuanhang Chemical Co. Ltd. P25 samples were heat treated in a conventional muffle furnace at 400, 500, 600, 700, 800,

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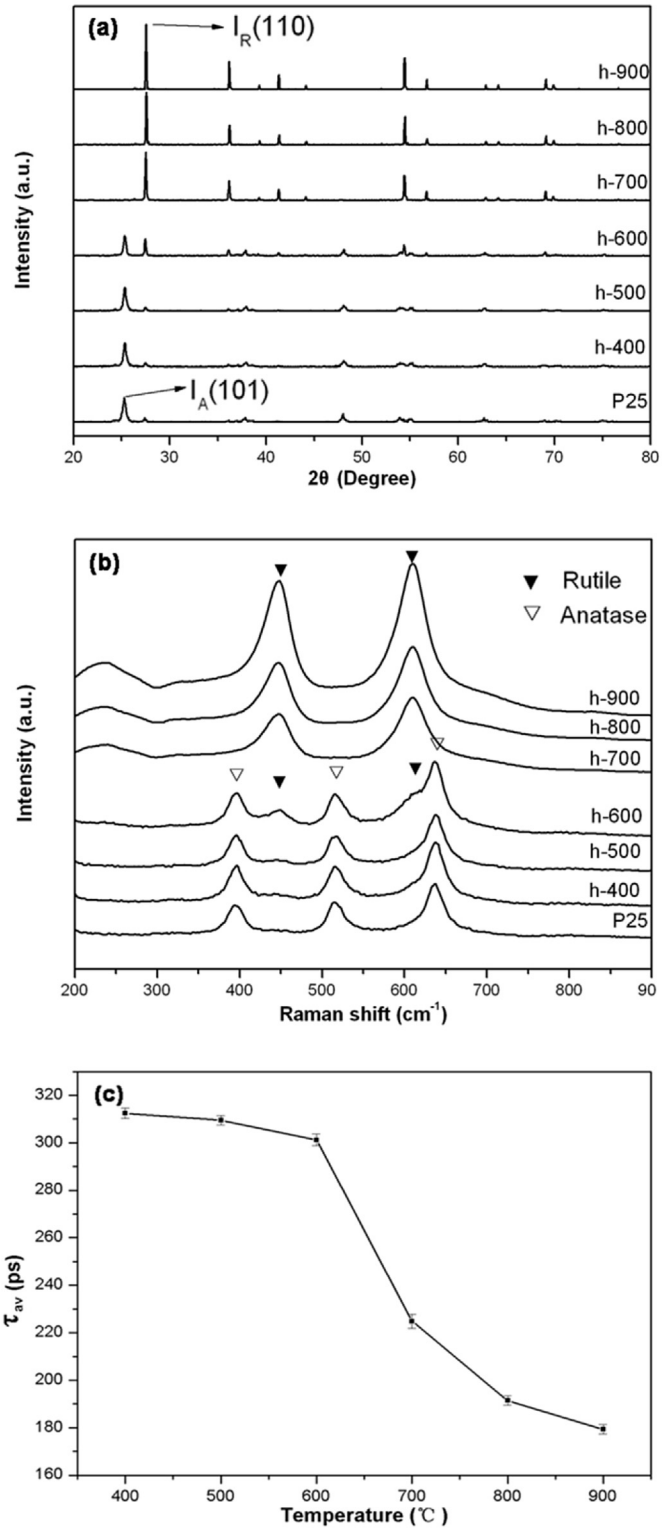

Fig. 1. XRD patterns (a), Raman spectra (b), and average positron lifetime τ_{av} (c) of titania containing different ratio of rutile.

and 900 °C for 3 h, respectively. The samples were denoted by h-x, where x represents calcination temperature.

Table 1
XRD, BET, and band-gap result of photocatalysts.

Samples	W_A[a]	W_R[b]	Crystallite size (nm)		S_{BET} (m^2/g)	BE (eV)[c]
P25	81.38%	18.62%	A 21.8	R 30.0	57.5	3.18
h-400	82.10%	17.90%	A 21.4	R 37.1	54.9	3.17
h-500	74.41%	25.59%	A 22.0	R 40.6	49.1	3.15
h-600	46.32%	53.68%	A 24.1	R 42.1	40	3.08
h-700	1.58%	98.42%	–	R 54.6	14.4	3.06
h-800	0.00%	100.00%	–	R 63.3	9.7	3.02
h-900	0.00%	100.00%	–	R 75.0	3.4	3.01

[a] W_A presents the phase content of anatase.
[b] W_R presents the phase content of rutile.
[c] BE is the band gap energy of samples.

2.2. Characterization

The X-ray diffraction (XRD) patterns were obtained by a Bruker D8 advance X-ray diffractometer using monochromatic Cu $K\alpha$ radiation (λ = 1.5406 Å) with an accelerating voltage of 40 kV and current of 40 mA. Horiba LabRAM HR Raman spectrometer, with excitation wavelength of 488 nm was used to characterize the structure of photocatalysts. The Bruuauer-Emmett-Teller (BET) surface area was measured on a Beijing JWGB JW-BK using N_2 adsorption at −196 °C. The positron annihilation lifetime spectra (PAL) were measured by a conventional fast-fast coincidence system with time resolution of 300 ps. Diffusive reflectance UV–vis absorption spectra (DRS) were collected on a Shimadzu UV-2550 spectrophotometer using integrating sphere attachment with barium sulfate as a reference. Temperature-programmed desorption (TPD) of CO_2 was measured on a TP-5080 adsorption instrument (Tianjin Xianquan Co.). The samples were pretreated in N_2 atmosphere at 400 °C to remove the surface adsorbate. The CO_2 adsorption process was taken at 25 °C, and CO_2 desorption was measured with heating rate of 10 °C/min.

2.3. Decolorization evaluation

The photoreaction was taken under a 160 W high-pressure Hg lamp (Shanghai Minghua Co.). The light focused onto a 200 mL of beaker filled with 100 mL of 20 mg/L CV solution and 0.10 g of photocatalyst. The beaker was put in a water tank to slow down the increase of temperature. Before irradiation, the suspension had been magnetically stirred in darkness for 1 h. Every 30 min, 1.6 mL of the suspension was collected and centrifuged to remove the photocatalyst. During the visible photoreaction, a cut-off filter (λ > 420 nm) was put on the top of the beaker. The concentration of CV was measured by the UV–vis absorption spectra.

3. Results and discussion

3.1. Photocatalysts characterization

XRD (Fig. 1a) and Raman spectra (Fig. 1b) were used to study the structures of different photocatalysts. In Fig. 1a, I_A (101) at 25.6° is the strongest peak of anatase, and I_R (110) at 27.7° is the strongest peak of rutile [19]. The phase content and particle size results (Table 1) suggest that the phase transition started at 500 °C and ended above 700 °C. From 500 °C to 900 °C, the crystallite size of rutile had grown up obviously from 40.6 nm to 75.0 nm. With the increase of heat treatment temperature, the intensities of the Raman peaks were getting stronger obviously (Fig. 1b), which is due to the formation of the dense structures during the heat treatment. The Raman shifts at 396, 515, and 636 cm^{-1} are attributed to the B_{1g}, A_{1g}, and E_g modes of anatase. The Raman shifts at 449 and

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