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Investigation on the transition crystal of ordinary rutile TiO₂ powder by microwave irradiation in hydrogen peroxide solution and its sonocatalytic activity

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Abstract

The transition crystal TiO₂ catalyst with high sonocatalytic activity was obtained utilizing the microwave irradiation in hydrogen peroxide solution. At the same time a series of affecting factors (microwave irradiation time, heat-treated time and heat-treated temperature) to prepare the TiO₂ catalyst on the sonocatalytic degradation of parathion were considered in this paper. The ultrasound of low power was used as an irradiation source to induce treated TiO₂ particles to perform catalytic activity. The results show that the sonocatalytic activity of the transition crystal TiO₂ powder is obviously higher than those of pure ordinary rutile and anatase TiO₂ powders. At last, the parathion in aqueous solution was degraded completely and became some simple inorganic ions such as NO_3^- , PO_4^{3-} , SO_4^{2-} , etc. The degradation ratio of parathion in the presence of the transition crystal TiO₂ catalyst attains nearly 80% within 60 min ultrasonic irradiation, while corresponding ones are only 65.23% and 53.88%, respectively, for pure ordinary rutile and anatase TiO₂ powders. © 2006 Elsevier B.V. All rights reserved.

Keywords: Transition crystal TiO₂ catalyst; Microwave irradiation; Sonocatalytic degradation; Parathion

1. Introduction

Since recent years a great deal of effort has been devoted to develop heterogeneous catalysts with high catalytic activities for solving environmental problems [1,2]. In many semiconductor materials, the TiO₂ powder is considered to be a very efficient catalyst [3] that, unlike other semiconductor materials, is non-toxic, stable, cheap and suitable for work using ultraviolet light as the energy source [4–6]. TiO₂ powder has been known to exist in three main polymorphic phases: rutile, anatase and brookite. All of them have the same fundamental structural units of octahedron, but their arrangements are different [7]. In general, the catalytic activity of anatase TiO₂ powder is better than that of rutile TiO₂ powder in the photocatalytic degradation reaction [8]. A recent study showed that a mixture of both anatase and rutile phases exhibits higher photoactivity as well as effective degradation in comparison with pure anatase or rutile catalysts [9]. However, the photocatalytic degradation must need ultraviolet light to induce TiO₂ powder, which cost lots of energy. Furthermore, in any case this method is not suit for the treatments of non- or low-transparent organic wastewater. In fact, it can avoid these disadvantages to utilize ultrasonic irradiation. The penetrating ability of ultrasound is very strong for any water medium and its penetrating depth can ordinarily attain to 15-20 cm [10]. Moreover, the ultrasound can usually be competent for catalyzing those chemical

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reactions that the ultraviolet and visible lights catalyze [11,12]. Hence, recently, the ultrasound has been used as irradiation source instead of ultraviolet and visible lights and many studies on the sonocatalytic degradation of various organic pollutants in the presence of TiO_2 powder have been reported [13].

Be similar to the photocatalytic degradation, the sonocatalytic activity also needs to be improved for high degradation effect. In present paper, the possibility of high sonocatalytic activity of transition crystal rutile TiO₂ powder by microwave-hydrothermal method was probed. The microwave-hydrothermal method is an emerging technique to prepare crystalline oxides in much shorter durations [14,15]. The advantages of microwave-hydrothermal process comparing to conventional hydrothermal methods are extremely rapid kinetics of crystallization, very rapid heating to the temperature of treatment and possible formation of new metastable phases. Fortunately, a novel and simple method for preparing transition crystal TiO₂ powder with high sonocatalytic activity has been realized in this paper. The experimental results show that the treated TiO₂ powder behaves high catalytic activities in the sonocatalytic degradation of parathion.



Molecular structure of parathion

2. Experimental

2.1. Materials and apparatus

Ordinary titanium dioxide powder (TiO₂, rutile and anatase, Haerbin Chemistry Reagent Company, China); Hydrogen peroxide (H₂O₂, AR, 30% content, Beijing Chemistry Reagent Corporation, China); Parathion (99.7% purity, Sittingbourne, Kent, USA). SX2-4-10 Muffle furnace (Great Wall Furnace Company, China); 101-1 oven (Shanghai Experiment Apparatus Company, China); WD750B micro-wave oven (2450 MHz, 800 W, Galanz Company, China); RINT-2700 XRD diffractometer (Rigaku Company, Japan); Phillp-EM400T transmission electron microscopy (Phillp company, Holand); LAMBDA-17 UV-vis spectrometer (Perkin–Elmer Company, USA); FT-IR (Perkin–Elmer Company, USA); KQ-100 Controllable Serial-Ultrasonics apparatus (40 kHz, 50 W, Kunshan apparatus Company, China); ICS-90 ion chromatogram (DIONEX Company, USA).

2.2. Preparation of the transition crystal TiO_2 sonocatalyst

The ordinary rutile TiO_2 powder was used as the titanium source and the preparation of transition crystal TiO_2 powder was carried out as following procedure. The ordinary rutile TiO₂ powder (2.0 g) and 30% hydrogen peroxide solution (20 mL) was added into reaction kettle, and then this mixed solution was treated under microwave irradiation for 5.0 min. The produced pale yellow slurry was filtrated and washed three times using purified water. Afterwards, these precipitates were dried at 100 °C for 60 min in oven in order to vaporize water, and then ground adequately to a fine powder to obtain dried samples. The dried samples were calcined in muffle furnace maintained at 400 °C for 30 min to obtain the transition crystal TiO₂ powder as a novel sonocatalyst.

2.3. Characterization of the transition crystal TiO_2 powder

In order to confirm the appearance of the anatase phase TiO₂, the XRD pattern of the treated TiO₂ powder was determined as shown in Fig. 1. The particle size (α) is approximatively counted by the Scherrer equation: $\alpha = 0.89\lambda/B_{1/2}\cos\theta$ (λ : wavelength of incident X-ray; $B_{1/2}$: width of half high peak; θ : diffraction angle). The proportion (β) of the rutile and anatase phases in the transition crystal TiO₂ powder was obtained according to the quantitative equation [16]: $\beta_{\rm R} = [1 + 0.8(I_{\rm A}/I_{\rm R})]^{-1}$ and



Fig. 1. XRD patterns of various TiO₂ powders: (a) transition crystal TiO₂ powder; (b) ordinary rutile TiO₂ powder.

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