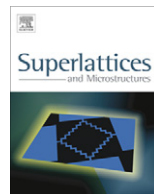




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Photocatalytic reactivity of Ce-intercalated layered titanate prepared with a hybrid method based on ion-exchange and thermal treatment

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ABSTRACT

Ce-intercalated layered titanate (CeTO) was synthesized through an ion-exchange process combined with thermal treatment. According to X-ray diffraction (XRD) analysis, the interlayer shrinkage, lattice cell contraction and lattice dislocation of CeTO occur during the ion-exchange process. The ion-exchange process lengthens the bond length of Ti–O in the *a*-axis direction, which results in a bathochromic shift of titanate in UV–vis spectrum. Calcination treatment intensifies the interlayer shrinkage and reduces the lattice dislocation via lattice rearrangement. The photocatalytic reactivity of CeTO is significantly improved with thermal treatment at 400 °C. EPR study shows that most of the intercalated cerium ions are oxidized to Ce⁴⁺ after intercalation, while thermal treatment changes little the chemical valence of cerium ion. It is suggested that lattice dislocation but not chemical valence of cerium ion plays an important role in determining the photocatalytic activity of CeTO. Further, the photocatalytic reactivity of CeTO is much higher than cerium doped sodium titanate prepared by solid-state reaction (SSR) method.

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1. Introduction

Titanates have attracted much attention due to their intriguing functional properties and wide applications in catalysis, sensorial applications, nanocomposites, and energy storage [1–5]. Titanates

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can be readily modified by intercalation of metal cations between the host layers [6,7], which always leads to significant changes in optical and magnetic properties, as well as photocatalytic activity [8,9]. For example, the absorption band edge of layered titanate had a significant bathochromic shift toward the visible light region by intercalation of Co^{2+} , which endowed titanate a visible light photocatalytic reactivity [10]. Gang et al. [8] claimed that the nickel species in the interlayer can provide active sites for proton reduction and cause fast diffusion of photoelectrons generated from titanate layers towards the nickel sites. Additionally, Co^{2+} modified trititanate nanotubes prepared by an in situ hydrothermal process were reported owning ferromagnetism at the room temperature [11].

Further, lanthanide cations began to attract special attention as (1) the special 4f orbit electron of lanthanide cations would influence the transfer of charge carriers [12], and (2) lanthanide cations always possess a strong adsorbability towards the organic substances [13]. Recently, some works began to appear about the photophysical and photochemical performances of layered titanate intercalated with lanthanide cations [14,15]. Viana et al. [16] synthesized Ce-intercalated titanate nanotubes (Ce-TiNTs), which exhibited a higher photocatalytic activity for degradation of Reactive Blue 19 dye as compared with the pristine titanate nanotubes. However, many preparation methods of lanthanide cations intercalated titanate involved solid-state reaction method (SSR) [17–19], polymerizable complex method (PC) [20] and sol–gel method [21], while the ion-exchange technique was relatively less used.

Although some works mentioned the original morphology of the raw layered titanate can be retained after ion-exchange reaction [7], intercalated metal cation may influence the crystal structure (lattice expansion or shrinkage) of layered titanate because of its discrepant size and/or charge towards the alkali metal ion. Hence, the relationship between the lanthanide cation intercalation and the crystal structure of layer titanate should be considered, which was however scarcely reported till now.

Three different methods have been mainly used for the guest cation intercalation into the interlayer of a layered oxide, which always possesses alkali cations only. One is the assembly of a host nanosheet with cations of interest in a solution due to the electrostatic principles, such as the electrostatic self-assembly deposition (ESD) [22] and layer-by-layer assembly technique (LBL) [23]. The second method involves a hydrothermal treatment of a pre-doped TiO_2 with definite wt.% of the desired cation in an alkali solution, that is, in situ doping [24,25]. However, the first method always encounters the contamination from the residual exfoliating agent, while the intercalated titanates prepared with the second method only present low photocatalytic reactivities [9,12,24,25]. The last method is the ion-exchange technique, in which the desired cation is substituted for alkali cation of the raw layered oxide [26–28]. Ion-exchange procedure can also be carried out in aqueous solution with the assistant of proton (H^+) [26,29–30]. In this work, sodium trititanate was prepared under hydrothermal condition and then ion exchanged with cerium ion. Sodium trititanate is a typical layer material and can readily be intercalated with various species via ion-exchange reaction [16]. The crystal structure and photocatalytic activity of as-prepared Ce-intercalated sodium titanate will be studied to analyze the possibility of photocatalytic reactivity modulation of intercalated titanate via metal ion intercalation.

2. Experimental section

2.1. Reagents and materials

$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, Na_2CO_3 , NaNO_3 and CeO_2 were purchased from Sinopharm Chemical Reagent and of AR. NaOH and HNO_3 were obtained from Lingfeng Chemical Reagent. P25 was obtained from Degussa. AO7 (Scheme 1, $\lambda_{\text{max}} = 483 \text{ nm}$) was obtained from Acros and used without further purification. Doubly distilled water was used throughout this work.

2.2. Synthesis of the layered sodium titanate

Layered sodium titanate was prepared by the hydrothermal method according to the Ref. [31]. In a typical synthesis, 2.00 g TiO_2 (Degussa P25) was dispersed in 75 mL of 10 M aqueous NaOH solution.

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