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Black Rutile TiO_2 Hydrogenated in Ultrafast Flow has Highly Stable Visible-Light Photocatalytic Capabilities

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Abstract

Black TiO_2, a new form of TiO_2 with excellent visible-light photocatalytic activity, can develop surface lattice disorder as a result of hydrogenation and the addition of Oxygen Vacancies (VO), but this TiO_2 is prone to failure because oxidation causes the concentration of surface VO to drop quickly and over short periods of time. In this study, VO-almost-concentrated black TiO_2 nanoparticles were created using an ultrafast hydrogen flow. These bulk VO increased the black TiO_2's ability to absorb visible light, decreased its bandgaps, and gave it incredibly strong stability. The presence of VO was confirmed by a number of characterization techniques, and degradation tests on Cr^{6+} or rhodamine B showed our material to have strong visible-light photocatalytic capability.

Keywords: ***oxygen vacancies, black rutile TiO2, and rapid hydrogen flow.***

Introduction

Since its photocatalytic property of degrading water was identified in TiO_2 in 1972, it has been extensively explored in regards to addressing environmental and energy challenges. However, due of its large bandgap and limited visible-light absorption, naturally occurring TiO_2 can only be stimulated by ultraviolet to form electron-hole pairs; this restriction makes TiO_2 a long way from practical application. Such techniques have been tried for reducing the bandgap of TiO_2 and improving its visible-light photocatalytic efficacy, such as doping and compounding with other semiconductor oxides. In 2011, Chen et al. announced a substance known as "black TiO_2," which instantly caught the public's attention due to its astoundingly decreased bandgap (1.54 eV) and dramatically enhanced visible-light photocatalytic activity. After being reduced in a hydrogen atmosphere with high pressure, these TiO_2 nanoparticles turned from white to black, but their phase remained TiO_2. Due to the significant visible light absorption of this form of TiO_2, a slightly lighter shade of TiO_2 with a similar mechanism was also referred to as black TiO_2 in later reports. Although there are numerous ways to make black TiO_2, hydrogenation is still the most popular method due to its high efficiency and simplicity in industrial production. The most widely accepted theory about the production mechanism of black TiO_2 was that Oxygen Vacancies (VO) played a key role in the formation of black TiO2. By adding donor energy levels into conduction bands,

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VO could alter the band topologies and visible-light absorption of black TiO_2. Ti^{3+} was frequently thought to appear concurrently with the generation of VO, and both could aid in the separation of photo generated electron-hole pairs. It has also been suggested that other flaws, such as surface hydroxyl groups, are to blame for the formation of black TiO_2, which exhibits excellent visible-light photocatalytic performance. Additionally, because these crystallographic flaws frequently accumulated on the material's surface, black TiO_2 typically had characteristic amorphous shells on its surface. While the photocatalytic performance of black TiO_2 long treated under various external danger factors (O2, H^+, OH, etc.) was still not researched, several researchers started to worry about and solve this problem. For instance, Lan et al. used an in-situ reduction technique to create as much Ti^{3+} inside the substance as feasible to stabilize black TiO_2. There are two types of VO, and they might be related to various locations, according to a study that was conducted at the same time. As the temperature of hydrogenation increased, the hues of black TiO2 gradually intensified, matching the spectra of diffuse reflectance in the UV-visible range (UV-Vis DRS, measured on Shimadzu UV-3600). An X-Ray Diffraction Spectrometer (XRD) using Cu K-X-ray was used to accomplish the phase analysis. EPR experiments were conducted at 77 K using a Bruker ELEXSYS-II E500 spectrometer. To emphasize the effect of pH on the material itself, the black TiO_2 particles were first treated with acid-base solutions rather than being immediately added to reaction solutions with different pH levels as is normal. After being aged for 18 months, the 800°C-H-TiO_2 powder was dispersed in various water for 180 hours while being pH-controlled aqueous solutions using HCl or NaOH. The pertinent outcomes and technical specifications were those previously mentioned. The photocatalytic activity of black TiO_2 was not affected by prolonged immersion in an acidic solution, although it was enhanced due to changes in surface states caused by protonation. An X-ray photoelectron spectrometer was used to gather information on surface hydroxyls and Ti states. Transmission electron microscopy (TEM, FEI Inspect F50) and scanning electron microscopy were used to capture microphotographs of the materials. Instead of being placed straight into reaction solutions with varying pH levels, as is customary, the black TiO_2 particles were first treated with acid-base to highlight the impact of pH on the material itself. The 800°C-H-TiO_2 powder was distributed in various water for 180 hours after being aged for 18 months using HCl or NaOH to control aqueous solutions with varying pH levels. The relevant results and process parameters were the same as those described above. Even though it was improved due to changes in surface states brought on by protonation, prolonged immersion in an acidic solution had no effect on the photocatalytic activity of black TiO_2.

Conclusion

In conclusion, this work successfully synthesized black rutile TiO_2 with exceptionally steady visible-light photocatalytic activity. The photocatalytic activity of 800°C-H-TiO_2 was the best, and its improved stability and visible-light photocatalytic activity may be attributed to the high concentration of bulk VO. The stability experiment for 800°C-H-TiO2 demonstrated that our black TiO_2 could be used steadily in acidic water and could sustain photocatalytic activity for 18 months.