

Synthesis Studies of Cu-TiO₂ for Photocatalytic CO₂ Reduction

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Abstract

Photocatalytic reduction of CO₂ to chemicals is a promising sustainable method to fight global warming using renewable energy. This process uses a photocatalyst activated by light to drive the reduction of CO₂. TiO₂ has been used as a benchmark photocatalyst for a long time, and it is easy to synthesise and affordable. Therefore, it is important to understand how to achieve high photocatalytic activity in CO₂ reduction using TiO₂ materials, particularly, by understand how catalyst synthesis conditions influence band gap and kinetic limitations.

The effect of synthesis conditions and the role of copper in the band gap of Cu-TiO₂ samples were studied. Samples were prepared by sol-gel in a semi batch reactor using titanium butoxide as precursor at different pH conditions. Copper was doped as aqueous Cu(NO₃)₂ either during the sol-gel procedure or afterwards by incipient impregnation. The performance of the samples was tested using reduction of 0.1 M bicarbonate to formate at 40 °C using an annular photo-reactor equipped with a 125 W mercury lamp. The samples were characterised by X-ray diffraction (XRD), scanning electron microscopy (SEM) and UV-Vis diffuse reflectance spectroscopy (DRS). It was found that the pH of the sol-gel process affected the crystalline phases, crystallite sizes and the band gap of the catalyst, revealing the important of controlling synthesis parameters. While commercial P25 TiO₂ was composed mostly of anatase and rutile, in-house TiO₂ prepared at pH of 0.4 showed predominantly rutile, but also anatase and brookite, while at pH of 1.5 no rutile diffraction patterns were observed. As the pH increased from 0.4 to 1.5, the anatase crystallite size decreased from 23.6 nm to 17.7 nm, with copper doping leading to a further reduction to 13.6 nm. The band gap of the samples prepared at pH 1.5 was measured at ~3.4 eV even after doping with Cu, while in the case of low pH conditions that band gaps were ~3.0 eV, revealing the influence of crystalline phases and crystallite size. Copper doping by impregnation led to double reaction rates comparatively to samples where copper was added directly during the gel synthesis.