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Organic-free hydrothermal synthesis of chalcopyrite CuInS₂ and its photocatalytic activity for nitrate ions reduction

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Abstract

We performed a facile and organic-free hydrothermal method (thiourea-oxalic acid co-molten system) to prepare chalcopyrite CuInS₂ nano-crystallite. Its intrinsic photocatalytic activities to nitrate reduction in aqueous solutions was investigated in comparison to bulk CuInS₂ prepared by annealing Cu₂S and In₂S₃ at 600-750 °C under vacuum. All samples show good and similar photocatalytic activities for nitrate reduction, for example, the conversion rate is about 2 times of that in a blank experiment. All CuInS₂ catalysts were stable after the photocatalytic reactions, indicated by the identical XRD patterns before and after the reaction.

Keywords: Chalcopyrite; CuInS₂; nanocrystalline materials; electron microscopy; photocatalysis; nitrate reduction.

1. Introduction

Chalcopyrite $CuInS_2$ is an important narrow band semiconductor ($E_g = 1.5 \text{ eV}$). People devoted a lot of efforts to develop morphology-controllable synthetic methods, however mostly involving organic reagents. [1-6] For example, the well-known hot-injection approach was reported in 2008 to prepare nearly monodisperse Cu-In-S nanocrystals (NCs) with either chalcopyrite or wurtzite structure. [1] A solvothermal route using triethylene glycol as the reaction medium was applied to prepare $CuInS_2$ hierarchical microarchitectures. [2] Monodisperse $CuInS_2$ NCs can be produced by injecting mixed metal-oleate precursors into hot organic solvents. Here we use a thiourea-oxalic acid mixed system to perform an

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