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Microwave-assisted synthesis of CdS intercalated $K_4Nb_6O_{17}$ and its photocatalytic activity for hydrogen production

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ABSTRACT

CdS intercalated $K_4Nb_6O_{17}$ (designated as $K_4Nb_6O_{17}$/CdS) composite photocatalysts were synthesized using a microwave-assisted synthesis. The composite particles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), X-ray fluorescence spectrometry (XRF), energy dispersive X-ray (EDS), ultraviolet–visible diffuse reflection spectra (UV–vis) and photoluminescence measurements (PL). The photocatalytic properties of these catalysts for hydrogen production were also investigated in the presence of Na_2S and Na_2SO_3 sacrificial reagents. The $K_4Nb_6O_{17}$/CdS catalysts synthesized using microwave irradiation were found to possess higher crystallinity than their counterparts, synthesized using conventional methods. The absorption edge of $K_4Nb_6O_{17}$ was shifted to the visible light region after the intercalation of CdS. Compared to the conventional synthesis method, the use of microwave irradiation sharply shortened the intercalation time. Furthermore, the $K_4Nb_6O_{17}$/CdS sample prepared via microwave irradiation exhibited higher activities for photocatalytic hydrogen production under both UV light and visible light irradiation, and the amounts of hydrogen produced were 265.95 mmol/(g cat) and 5.68 mmol/(g cat) after 3 h of irradiation, respectively. The mechanism of separation of the photo-generated electrons and holes at the $K_4Nb_6O_{17}$/CdS composite was discussed.

1. Introduction

The decomposition of water into hydrogen and oxygen by photoinduced processes employing a semiconductor irradiated with light can be used for sustainable solar energy conversion, and has been investigated in recent years [1–4]. Layered semiconductors are a group of compounds which demonstrate high activities for photocatalytic water splitting, and some of these semiconductors such as $K_4Nb_6O_{17}$ [5], $K_2Ti_4O_9$ [6] and $K_2La_2Ti_3O_{10}$ [7] have been reported to facilitate hydrogen production using photo-processes.

$K_4Nb_6O_{17}$ is a two-dimensional layered compound, which is formed by the connection of octahedral units of NbO_6 and oxygen bridging atoms. $K_4Nb_6O_{17}$, like other layered compounds, cannot exhibit any photocatalytic activity under visible light irradiation, due to its relatively large band gap (about 3.2 eV [8]). The intercalation of nano-semiconductor particles with narrow band gap in the layered niobates of $K_4Nb_6O_{17}$ can extend the absorption edge into the visible light region, and hence improve the photocatalytic activity by using these narrow band gap semiconductors as sensitizers. Some researchers reported the successful improvement of photocatalytic activity of $K_4Nb_6O_{17}$ under visible light through the intercalation of nano-sized CdS [9,10]. The existence of nano-sized CdS particles in the layered space of $K_4Nb_6O_{17}$ compounds cannot only prevent the photo-corrosion of these nano-particles, but can also promote the efficiency of separation of photogenerated elections and holes, facilitating the photocatalytic activity of hydrogen evolution under visible light irradiation.

The preparation of CdS intercalated $K_4Nb_6O_{17}$ via conventional methods can take several days to several weeks [9,11] due to mass transfer limitations. Additionally, the preparation process is difficult to control, and the crystal structure of $K_4Nb_6O_{17}$ could be consequently destroyed [11,12]. It has been reported that increasing the crystallinity of nano-TiO_2 could enhance photocatalytic activity [13], because of higher migration rates of photo generated carriers in the crystals. Additionally, methylthionine chloride intercalated $K_4Nb_6O_{17}$ catalysts were prepared in the literature, and the results obtained indicated that the photocatalytic activity of the catalysts could be effectively improved by selecting $K_4Nb_6O_{17}$ with higher crystallinity [14].

Microwaves consist of electromagnetic waves whose wavelength varies from 1 mm to 1 m, and application of this electric field causes the vibration of dipolar molecules. An electromagnetic field changes its direction at a frequency of hundreds of millions or even billions times per second. Since the dipolar molecules cannot keep up with the rate of electromagnetic field conversion, friction and the consequent production of heat ensues. In this sense, microwave energy is extremely efficient in the selective heating of materials. Compared to the conventional treatment, this has the effect of

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