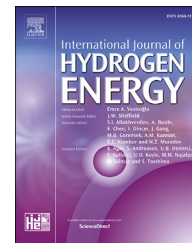




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Construction of ternary hybrid layered reduced graphene oxide supported g-C₃N₄-TiO₂ nanocomposite and its photocatalytic hydrogen production activity

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ABSTRACT

Reduced graphene oxide (rGO) supported g-C₃N₄-TiO₂ ternary hybrid layered photocatalyst was prepared via ultrasound assisted simple wet impregnation method with different mass ratios of g-C₃N₄ to TiO₂. The synthesized composite was investigated by various characterization techniques, such as XRD, FTIR, Raman Spectra, FE-SEM, HR-TEM, UV–vis DRS Spectra, XPS Spectra and PL Spectra. The optical band gap of g-C₃N₄-TiO₂/rGO nanocomposite was found to be red shifted to 2.56 eV from 2.70 eV for bare g-C₃N₄. It was found that g-C₃N₄ and TiO₂ in a mass ratio of 70:30 in the g-C₃N₄-TiO₂/rGO nanocomposite, exhibits the highest hydrogen production activity of 23,143 μmol g⁻¹h⁻¹ through photocatalytic water splitting. The observed hydrogen production rate from glycerol-water mixture using g-C₃N₄-TiO₂/rGO was found to be 78 and 2.5 times higher than g-C₃N₄ (296 μmol g⁻¹ h⁻¹) and TiO₂ (11,954 μmol g⁻¹ h⁻¹), respectively. A direct contact between TiO₂ and rGO in the g-C₃N₄-TiO₂/rGO nanocomposite produces an additional 10,500 μmol g⁻¹h⁻¹ of hydrogen in 4 h of photocatalytic reaction than the direct contact between g-C₃N₄ and rGO. The enhanced photocatalytic hydrogen production activity of the resultant nanocomposite can be ascribed to the increased visible light absorption and an effective separation of photogenerated electron-hole pairs at the interface of g-C₃N₄-TiO₂/rGO nanocomposite. The effective separation and transportation of photogenerated charge carriers in the presence of rGO sheet was further confirmed by a significant quenching of photoluminescence intensity of the g-C₃N₄-TiO₂/rGO nanocomposite. The photocatalytic hydrogen production rate reported in this work is significantly higher than the previously reported work on g-C₃N₄ and TiO₂ based photocatalysts.

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Introduction

Hydrogen is considered as a renewable and alternative fuel due to its earth abundance, zero emission and high combustion energy [1]. The solar energy conversion to hydrogen via photocatalytic water splitting is one of the promising methods to produce hydrogen because of low cost and high sustainability features of the reaction system [2]. Since 43% of the sunlight is in the visible region, it makes sense to have a visible light active photocatalyst. Most commercially available photocatalyst are either having band gap in the UV region or suffer from rapid recombination of electron – hole pairs, photo-corrosion and photo-instability [3]. In the past decades, considerable efforts have been made to overcome these challenging issues by designing a semiconductor-semiconductor, semiconductor-metal or semiconductor-carbon junction [4].

Graphitic carbon nitride ($g\text{-C}_3\text{N}_4$), a low cost, nontoxic, highly stable semiconductor with band gap energy in the visible range, is one of the potential candidates for photocatalytic hydrogen production [5,6]. Unlike other organic π -conjugate materials, $g\text{-C}_3\text{N}_4$ is a crystalline material due to its lamellar structure which helps it to facilitate the charge transfer [7,8]. However, bulk $g\text{-C}_3\text{N}_4$ shows low photocatalytic hydrogen production, due to low specific surface area and rapid recombination rate of electron-holes pairs [9,10]. On the other hand, ultrathin $g\text{-C}_3\text{N}_4$ nanosheets and quantum dots are known to show excellent photocatalytic water splitting activity under visible light irradiation [11,12].

Coupling of $g\text{-C}_3\text{N}_4$ nanosheets with other metal oxides such as N, S– $\text{TiO}_2/g\text{-C}_3\text{N}_4$ [13], $g\text{-C}_3\text{N}_4/m\text{-Ag}_2\text{Mo}_2\text{O}_7$ [14], $g\text{-C}_3\text{N}_4/\text{InVO}_4$ [15] and $g\text{-C}_3\text{N}_4/\text{NiFe-LDH}$ [16] has shown to increase photocatalytic hydrogen evolution efficiency. Moreover, loading of noble metals on the surface of $g\text{-C}_3\text{N}_4$ such as Pt-CF/ $g\text{-C}_3\text{N}_4$ [17], Ag/ $g\text{-C}_3\text{N}_4$ [18] etc. has shown to be one of the ways of suppressing rapid recombination of photo-generated electron-hole pairs. But due to high cost of noble metals, it is highly required to explore low cost cocatalyst for developing highly efficient $g\text{-C}_3\text{N}_4$ based visible light photocatalyst.

TiO_2 is an efficient cocatalyst and alternative to the high cost noble metals for photocatalytic hydrogen production due to its low-cost, photoactivity, nontoxicity and excellent photostability. However, the wide band gap of TiO_2 of $\sim 3.18\text{eV}$ and rapid recombination of electron-holes pairs hinder its photocatalytic hydrogen evolution activity [19,20]. The hybridization of $g\text{-C}_3\text{N}_4$ with TiO_2 has shown to reduce the recombination rate of electron-hole pairs, resulting in an enhanced photocatalytic activity [21]. Chai et al. observed that the photocatalytic hydrogen production activity of $g\text{-C}_3\text{N}_4\text{-Pt-TiO}_2$ was almost 14 and 36 times higher than bare $g\text{-C}_3\text{N}_4$ and Pt– TiO_2 , respectively [22]. Gholipour et al. reported that the synthesis of $g\text{-C}_3\text{N}_4$ nanosheets and Titanate nanodisks via facial wet impregnation method enhanced the photocatalytic hydrogen production activity by 2.6 -folds high compares to bare $g\text{-C}_3\text{N}_4$ [23].

Graphene, sp^2 hybridized carbon mono layer, has attracted tremendous interest due to its large surface area and high electron mobility. It is considered as viable alternative to the

expensive noble metals as cocatalyst for charge carrier separation and transfer. Graphene can be produced with a relatively perfect structure and properties by chemical vapour deposition or epitaxial growth methods [24]. However, pure graphene is hydrophobic and expensive to produce on large scale [25]. While in comparison, graphene oxide (GO) is hydrophilic in nature and facilitates the construction of microstructures in a stable colloidal solution. GO can be produced on large scale from graphite by chemical methods and it can be reduced to produce pristine graphene like material called as reduced graphene oxide (rGO).

$g\text{-C}_3\text{N}_4$, rGO and TiO_2 are some of the most promising materials for photocatalytic hydrogen production activity. Recently, Meina Huan et al. have reported that coupling of the nanocomposite $g\text{-C}_3\text{N}_4/(\text{anatase})\text{TiO}_2$ with rGO by one step solvothermal method, improves the photocatalytic dye degradation activity of $g\text{-C}_3\text{N}_4/\text{TiO}_2$ nanocomposite [26a]. However, the hydrogen production rates reported for bare $g\text{-C}_3\text{N}_4$ [22], TiO_2 [26b], P25 (degussa) [26c], $g\text{-C}_3\text{N}_4/\text{TiO}_2$ [23], TiO_2/rGO [26b] nanocomposites in the literature are relatively low. Compare to bare anatase and rutile phase, anatase-rutile mixed phase TiO_2 is known to yield better hydrogen production activity. In the present study, anatase-brookite mixed phase TiO_2 was prepared by a hydrolysis and peptization method and used to prepare a hybrid $g\text{-C}_3\text{N}_4\text{-TiO}_2/\text{rGO}$ nanocomposite for photocatalytic hydrogen production activity under UV–vis irradiation in water-glycerol mixed solution. The photocatalytic hydrogen production activity of $g\text{-C}_3\text{N}_4\text{-TiO}_2$ nanocomposite was found to be $19,610\ \mu\text{mol g}^{-1}\text{ h}^{-1}$; a remarkably higher value than reported before. Moreover, incorporation of rGO in the $g\text{-C}_3\text{N}_4\text{-TiO}_2$ nanocomposite further enhanced the hydrogen production rate by an additional $3533\ \mu\text{mol g}^{-1}\text{ h}^{-1}$.

Experimental details

Materials and characterization

Melamine (99.0%) and graphite powder (99.9995%) were purchased from Alfa Aesar. Titanium tetraisopropoxide (TTIP) (97.0%), isopropanol (99.0%) and glycerol were purchased from Sigma Aldrich, India. All other chemicals and solvents were used without any further purification and deionized (D.I) water was used in all the experiments.

The crystal structure and phase purity were analysed by X-ray diffraction (PANalytical X'pert powder diffractometer) using Cu $K\alpha$ radiation ($\lambda = 1.5406\ \text{\AA}$) in the range of $5^\circ - 90^\circ$ at room temperature. The morphology of the as prepared samples was examined by field emission-scanning electron microscopy (FEI Quanta FEG 200 HR-SEM). UV–visible absorption spectra and diffuse reflectance spectra (DRS mode) were obtained using UV-2600 spectrophotometer (Shimadzu) in the range of 300–800 nm. Fourier transform infrared spectroscopy (FTIR) was obtained using Perkin Elmer-USA. Transmission electron microscopy (TEM) images were obtained from Philips CM 200 transmission electron microscope with a LaB6 filament and equipped with an ultrathin objective lens and CCD camera. HRTEM images were recorded by using JEOL 3010

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