

Effect of N-doping on visible light activity of TiO₂–SiO₂ mixed oxide photocatalysts



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ABSTRACT

The present report deals with the effect of nitrogen doping on TiO₂–SiO₂ mixed oxide photocatalysts. Advantageous effect of mixing SiO₂ with TiO₂ and subsequent N doping on the visible light photocatalysis is discussed. Hydrazine is used as the nitrogen doping source. Mixed Oxide to hydrazine ratio is varied from 1:2 to 1:8 to study the hydrazine concentration effect on the surface and photocatalytic properties. Presence of silica increases the surface area and thermal stability of anatase phase whereas nitrogen doping enhances the visible light absorption. Presence of substitutional nitrogen and oxygen vacancy in TiO₂ lattice is due to the use of hydrazine which is also a reducing agent. Material prepared with 1:6 hydrazine ratio (TiSi-6N) shows surface area of 260 m²/g and light absorption up to 520 nm. Under visible light only TiSi-6N is able to completely reduce Se(VI) into its elemental form within 90 min of reaction.

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1. Introduction

Titanium dioxide (TiO₂) is one of the important materials used in photocatalysis and photovoltaics because of its low cost, nontoxic and noncorrosive nature [1–4]. It is well understood that photocatalytic efficiency of TiO₂ is greatly affected by the crystallinity, surface area and band gap. Generally anatase phase of titania with a band gap of 3.2 eV shows better photocatalytic activity. However, it is metastable and converts to less active rutile phase on heat treatment. Moreover, wide band gap limits its activity in UV range only. Therefore, last two decades have witnessed a great deal of research on band gap reduction so as to extend the absorption in visible range, which accounts for more than 40% of the solar spectrum [4–6]. Chemical modification of TiO₂ extends its activity in visible range, which can be enhanced further by controlling its morphology. In this context it was observed that metal or non-metal doping increases the visible light absorption to a large extent [5]. Indeed the seminal work of Asahi et al. on band gap reduction of TiO₂ by N doping stimulated the research on non-metal doped TiO₂ [6]. Afterwards, significant works are reported on investigation of the structural, electronic

and optical properties of N-doped TiO₂ by various authors [5,7]. Because of its comparable ionic size, N can be easily introduced in TiO₂ lattice. Various N-containing molecules such as N₂, NH₃, N₂H₄, HNO₃, NH₄OH, NH₄Cl, NH₄NO₃ and NH₂CONH₂ have been used in gaseous, liquid or solid phase [6–12]. However, nature and amount of the N doping vary with the N source chosen and synthetic methodology. Notwithstanding, in most of the cases N-doped TiO₂ loses its catalytic activity to great extent on increasing the calcination temperature, because of phase transformation and loss of N from the TiO₂ lattice.

On the other hand maintaining high surface area even after required heat treatment is important for a catalyst. In particular material may be subjected to heat treatment at or above 500 °C for effective non-metal doping which mostly reduce the surface area of titania material. In this context, the introduction of a second metal oxide (SiO₂, ZrO₂ etc.) has been proved to be an effective route to maintain appreciable surface area and improve the photocatalytic activity of TiO₂ [13–17]. The presence of SiO₂ in the N-doped TiO₂ increases the surface hydroxyl groups, suppresses the formation of rutile phase and inhibits the crystal growth during calcination [18–20]. In addition presence of SiO₂ can increase the surface acidity thus facilitating better photocatalytic activity [15,17]. This prompted us to have detail investigation of N doped TiO₂–SiO₂ systems in comparison to pure TiO₂. Previous reports have indicated that a low amount of (5–10%) SiO₂ addition is optimum for better photocatalytic activity of TiO₂ [13–15,18,20]. In

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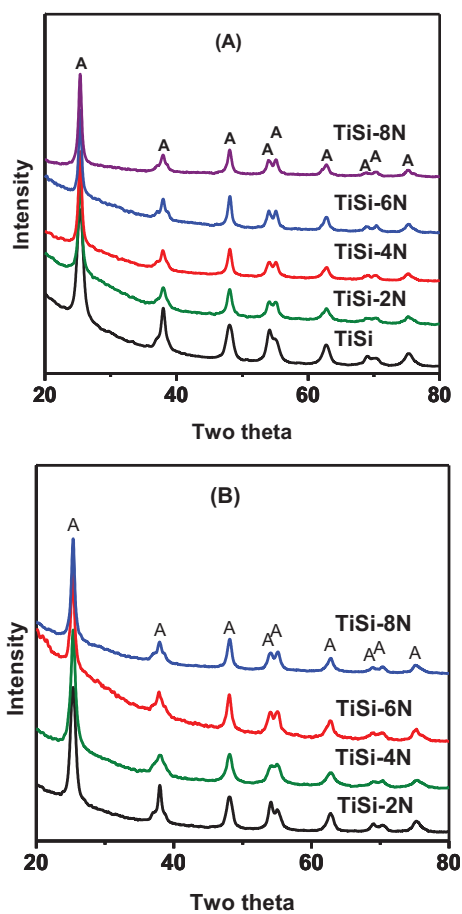


Fig. 1. XRD of N-doped TiSi calcined at (a) 400 °C and (b) 600 °C (A: Anatase).

previous work use of hydrazine during synthesis as a source of N resulted in high surface area material with the signature of Ti^{3+} [21]. So here different concentration of hydrazine is used to substantiate its effect on physical–chemical properties, porosity and photocatalytic activity under visible light.

Photocatalysts are generally used for oxidative decomposition of various pollutants. However, this can be used in reductive removal of different toxic elements [13,14,21]. Selenium is essential for humans and animals in trace amount but is very toxic at higher concentrations. Selenium is one of the toxic elements generally found in the waste water close to different mines. This creates problems in the

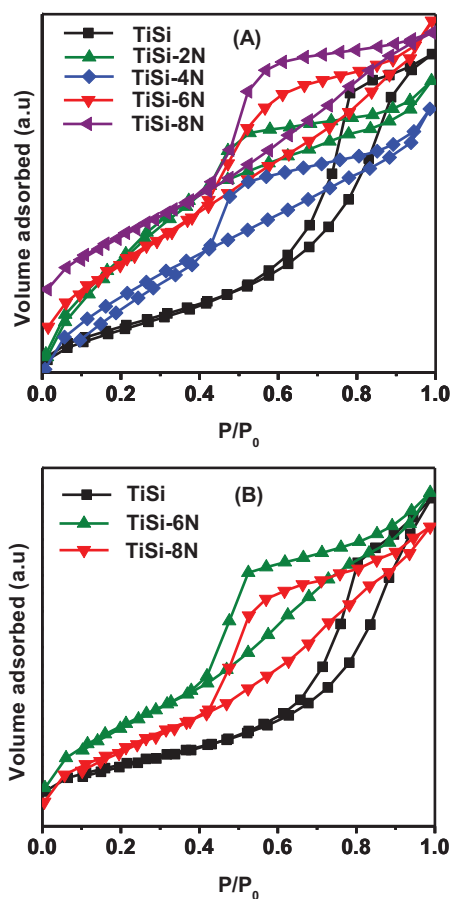


Fig. 3. N_2 adsorption-desorption isotherm of materials calcined at (a) 400 °C and (b) 600 °C.

water systems for human use in different parts of the world. Keeping this in view in this paper we report the synthesis of high surface area N-doped TiO_2 - SiO_2 mixed oxide photocatalyst for the improved photoreduction of Se(VI) under visible light.

2. Material and methods

2.1. Synthesis

Titanium silicon (TiSi) mixed oxide with 90:10 wt% was synthesized by controlled hydrolysis of titanium isopropoxide

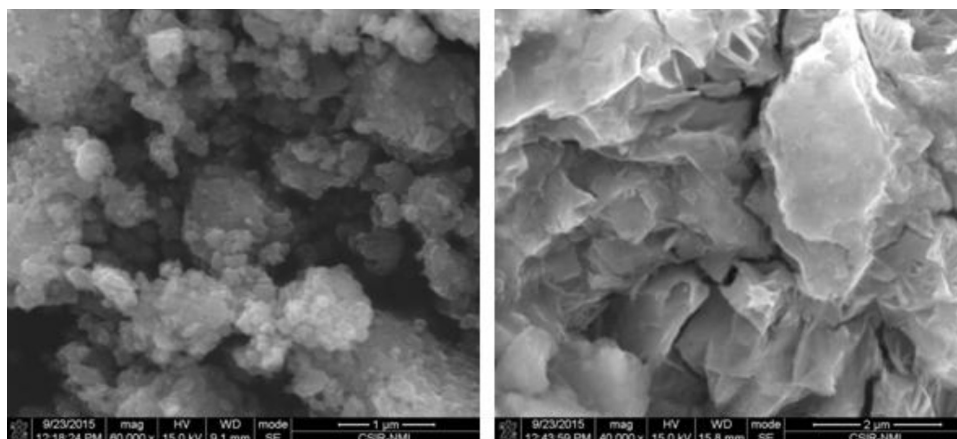


Fig. 2. SEM image of TiSi-6N (a) as prepared and (b) after selenate reduction reaction.

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