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Synthesis and characterization of zinc oxide nanorods and its photocatalytic activities towards degradation of 2,4-D

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ABSTRACT

Semiconductor zinc oxide nanorods (NRZnO) were prepared by sol-gel technique using zinc acetate as the precursor and ammonia as the precipitating agent. The prepared photocatalyst were characterized by X-ray diffraction (XRD), Fourier Transform Infrared (FTIR), UV–Visible diffuse reflectance spectroscopy (UV–Vis–DRS), X-ray photoelectron spectroscopy (XPS), Field Emission-Scanning Electron Microscopy (FE-SEM), Atomic Force Microscopy (AFM), High Resolution-Tunneling Microscope (HR-TEM), Brunauer, Emmett and Teller (BET) and electron paramagnetic resonance spectroscopy (EPR) analysis. Particle size of the prepared photocatalyst was established by XRD and FE-SEM analysis. The morphology and the formation of uniform NRZnO was controlled by the temperature, entire concentration of precursors, duration of aging process. High surface roughness and porosity confirmed by AFM analysis. Band gap energy of the synthesized photocatalyst (3.2 eV) was determined by using diffuse reflectance spectroscopy. The in-situ production of OH radicals by the prepared photocatalyst was confirmed by electron paramagnetic resonance spectroscopy (EPR) spin trapping technique. The photocatalytic activity of prepared NRZnO was evaluated by photo degradation of 2, 4-dichlorophenoxyacetic acid (2, 4-D) under UV and visible light irradiations. Experimental parameter such as effect of pH, catalyst dosage, initial 2, 4- D concentrations and addition of different electrolytes on the degradation of 2, 4-D was also studied in detail. Neutral pH was found to be the optimum and catalyst dosage of 30 mg/10 ml resulted in higher percentage of degradation. The photo degraded samples were analyzed by chemical oxygen demand (COD) analysis, UV–Visible spectroscopy. Reusability of the prepared photocatalyst was tested upto three cycles without affecting its performance. The experimental shown the rate of degradation follows pseudo-first order rate kinetics with respect to 2, 4 D.

1. Introduction

An increase in industrialization has resulted in large amounts of hazardous wastes being discharged into aquatic eco system (Valavanidis et al., 2006; Sharma et al., 2007). In recent years, there has been a growing interest on research and development in the area of heterogeneous photocatalytic water purification processes due to its effectiveness in degrading toxic organic compounds. Recently photocatalysis has been considered as one of the most important eco-friendly and clean chemical technology for environmental application (Anastas et al., 2000). Conventional methods such as solvent extraction, adsorption and chemical oxidation are often employed for environmental remediation but they suffer from serious limitations and disadvantages (Curridal et al., 2003). Heterogeneous photocatalytic oxidation processes are currently being considered as a better technique for the oxidation of organics in comparison to other conventional methods (Baruah and Dutta, 2009; Sugunan and Dutta, 2008). Hence, semiconductor photocatalytic technology has been employed as a tool to overcome all these drawbacks. Semiconductor photocatalysts for example, TiO_2, ZnO, WO_3, SnO_2 and CeO_2 etc are often employed for the various applications in the field of photocatalytic remediation, water splitting, sensors and solar cells (Gomez-Solís et al., 2015; Law et al., 2005; Fenoll et al., 2012; McCue and Ying, 2007; Miseki et al., 2010; Coronado et al., 2002).

As reported earlier, ZnO is considered to be a better photocatalyst than TiO_2 in the photocatalytic degradation of organic pollutants under UV and visible light irradiations (Chakrabarti and Dutta, 2004; Hong et al., 2006). As one of the established semiconductor photocatalyst, zinc oxide (ZnO) is used as a photocatalyst due to its wide band gap energy (3.2 eV), lower cost, higher activity and ecofriendly properties (Wang et al., 2012; Hariharan, 2006). The wide band gap energy of this photocatalyst retards the recombination of electron-hole pairs and enhances the photocatalytic activity (De Lasa et al., 2005). Different morphology of ZnO nanostructures have been synthesized such as

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hollow porous micro-flakes, flocky spheres, rose like structure, umbrella- like, castle like, hexagonal cones, fibers, nano nails needles, nanorods, nanowires, stars, webs and tetrapods (Duan et al., 2007; Fu et al., 2008; Xu et al., 2008; Du et al., 2007; Wang et al., 2006; Ren et al., 2007; Liu et al., 2008; Umar et al., 2008; Hu et al., 2004; Wu et al., 2004; Yang et al., 2006; Wang et al., 2005; Uhm et al., 2007; Lin et al., 2009). Among these morphologies, nanorod has more concern, because of their promising application to various fields such as light-emitting diode (Kim et al., 2011), gas and chemical sensors (Foo et al., 2013a, Foo et al., 2013b, Foo et al., 2013c), ultraviolet (UV) detector (Chai et al., 2009; Foo et al., 2013a, 2013b, 2013c), solar cell (Guillen et al., 2011; Matsubara et al., 2003) and biomolecular sensors (Fulati et al., 2010; Ali et al., 2010). Various methods have been investigated for the preparation of NRZnO through metal-organic chemical vapor deposition (Lee et al., 2004), metal-organic vapor phase epitaxy (Park et al., 2002), thermal evaporation (Grabowska et al., 2005) and thermal chemical vapor deposition (Hirate et al., 2007). In hydrothermal method templates are used for the preparation of NRZnO growth on Si substrate (Polsongkrama et al., 2008). All these methods are intricate and use high temperature with cumbersome experimental procedures. Among these methods, sol-gel method has attracted considerable attention because of its unique advantages such as simplicity, low cost, low temperature, high yield and controllable particle size. At low temperature (75 °C) and at low pH, the NRZnO are slowly formed (Simon et al., 2007). Thus, this method is considered to be a better technique for large scale production of uniform NRZnO materials without the use of templates.

Hence, the present work is focused on the preparation of nanorod ZnO (NRZnO) by simple sol-gel method and investigation on its catalytic activities were evaluated for the degradation for a model organic pollutant herbicide such as 2, 4-D under UV and visible light irradiations. The morphology and the formation of uniform NRZnO were controlled by temperature, concentration of precursors, duration of aging process and deposition time. The prepared NRZNO were characterized by XRD, UV-DRS, FT-IR, AFM, FE-SEM, XPS, BET, EDAX, HR-TEM, SAED and EPR techniques. The photodegraded samples were analyzed by UV–Visible spectroscopy, ESI-Mass and chemical oxygen demand (COD) analyses.

2. Experimental

2.1. Materials and methods

2, 4-Dichlorophenoxyacetic acid and zinc acetate ($Zn(OAc)_2$) were obtained from S.D. Fine Chemicals Ltd., India. The other chemicals used in these studies are Sodium hydroxide, Hydrochloric acid, Sulphuric acid (Merck), Potassium dichromate (Extra pure AR, SRL), Ferroin solution (AR, SD Fine Chemicals ltd), Ferrous ammonium sulfate (Extrapure AR, SRL), Silver sulfate (Merck). All the solutions were prepared by using deionised water.

The nanorod ZnO was synthesized from its precursor through a simple sol-gel method. The highly UV and visible active catalyst was

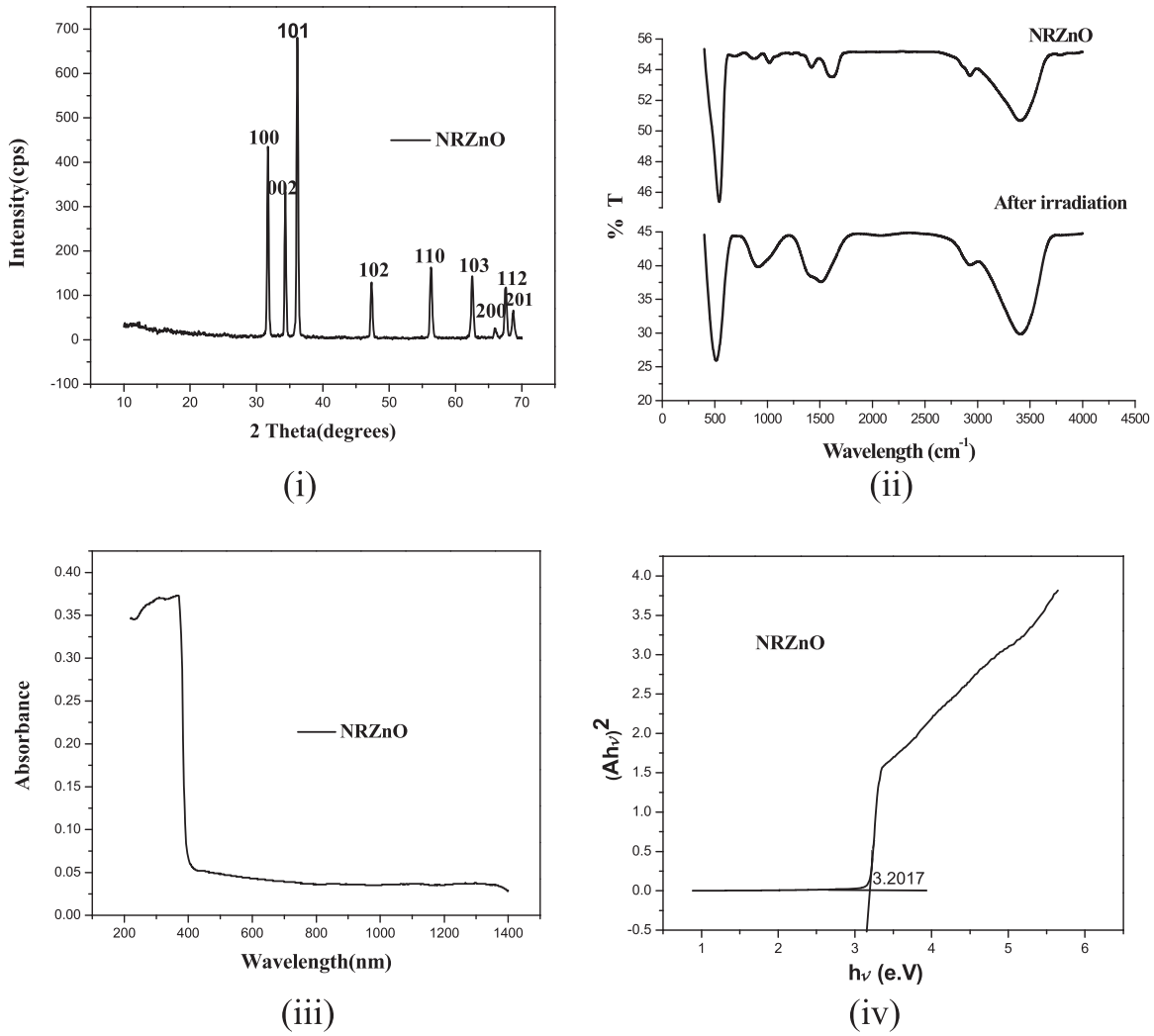

Fig. 1. XRD patterns of (i) NRZnO (ii) FTIR spectrum of NRZnO (iii) UV-DRS spectrum of NRZnO and (iv) shows a plot of $(Ahv)^2$ vs hv for the determination of band gap of NRZnO.

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