

Supplementary File

A comparative study on the photo-removal of few selected priority organic pollutants in aqueous suspension based on vanadium-doped-ZnO/MWCNT

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2. Experimental

2.1. Reagents and chemicals required

The precursors used to synthesize the photocatalyst, such as zinc acetate dehydrate, vanadium oxide, multiwalled carbon nanotubes and pollutants like 5-nitroisophthalic acid, allopurinol, and chloramines-T were obtained from Sigma Aldrich. The water used in all the experiments was double distilled. The photocatalyst TiO₂, Degussa P25 (Degussa AG), was used in most of the experiments, whereas other TiO₂ powders, namely Hombikat UV100 (Sachtleben Chemie GmbH) and PC500 (Mellinnum inorganic chemicals) were used for comparative studies. Degussa P25 consists of 75% anatase and 25% rutile with a specific BET surface area of 50 m²g⁻¹ and a primary particle size of 20 nm¹. Hombikat UV100 consists of 100% anatase with a specific BET surface area >250 m²g⁻¹ and a primary particle size of 5 nm². The photocatalyst, PC500 has a BET surface area of 287 m²g⁻¹ with 100% anatase and primary particle size of 5-10 nm.

2.2. Synthesis of ZnO nanorod

ZnO nanorod was prepared by the direct thermal decomposition of zinc acetate dihydrate as reported in previous literature with slight modification³. In order to prepare the substance described above, 10 g of zinc acetate dihydrate were placed in an alumina crucible, sealed with a lid, and then heated in a muffle furnace at a temperature of 400 °C for 4 h. The obtained material

was subjected to filtration and washing in distilled water in order to rid it of any undesired impurities before being dried overnight at a temperature of 90 °C.

2.3. Synthesis of V@ZnO/MWCNT nanocomposite

A well-known impregnation procedure was employed to obtain a 6 mol% V@ZnO/MWCNT nanocomposite. In a standard synthesis technique, the required quantity of MWCNT was dissolved in 50 mL water and sonicated for 30 min followed by stirring. Then 2 g of ZnO rod was dispersed in this solution and heated at 80 °C for 30 min. Now the 6 mol% of V₂O₅ was added to the above mixture to get 6 mol% V@ZnO/MWCNT. The whole mixture was heated at 80 °C for 2 h. Then washed with H₂O and ethanol for several times and dried to get final nanocomposite.

2.4. Material characterization

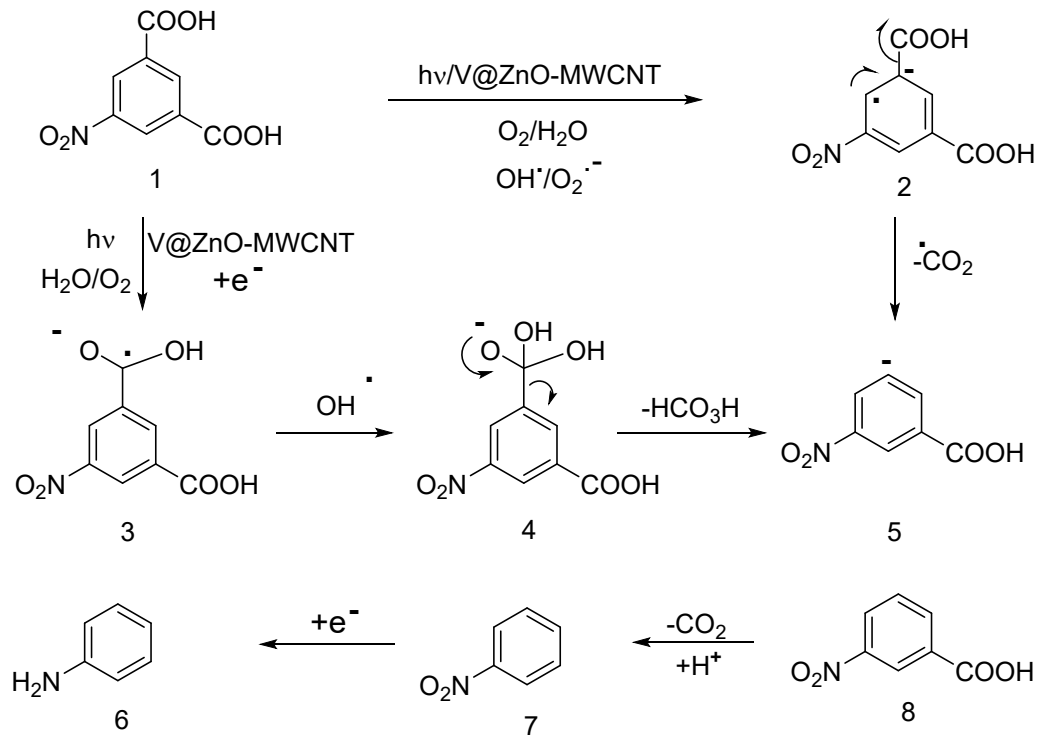
The synthesized samples were characterized using standard analytical techniques such as XRD, UV–vis DRS, FTIR spectroscopy, TEM, EDS and XPS. The crystal structure and phase of prepared samples were analyzed using an X-ray beam diffractometer (Smart Lab SE) using graphite monochromatic copper radiation (Cu K α radiation, 1.54 Å) operating at a voltage of 30 kV, a current of 15 mA in the 2 θ range between 5⁰ and 80⁰, and an output speed of 10⁰ per minute. The infrared (IR) spectra of the synthesized compounds was recorded using an FTIR spectrophotometer (PerkinElmer Spectrum 2), with KBr as a standard. To gain a deeper insight into the optical characteristics of the samples, we measured them with a UV–vis diffuse reflectance spectrophotometer (model number PerkinElmer Lambda 35). The morphology and purity of the synthesized materials were determined using SEM (JEOL JSM-6510LV) and TEM (PHILIPS CM 200). The elemental composition and surface chemical state of the composite material were studied by XPS (AXIS Supra) with Al K α – 1486.6 eV radiation.

2.5. Degradation procedure

The solution of the compounds of desired concentration was prepared in water and placed in an immersion well photochemical reactor made of pyrex glass with a magnetic stirring bar, water circulating jacket and an opening for the supply of molecular oxygen was used. A detailed description of the photochemical reactor is documented in our previous paper ⁴. The desired aqueous solution of the compound was taken in the vessel and photocatalyst (1 gL^{-1}) was added followed by sonication and bubbling of air for at least 15 minutes, which was then allowed to stir in the dark for 30 min to ensure the adsorption-desorption in equilibration. The pH was adjusted by adding a dilute aqueous solution of NaOH or HNO₃. A 125 W medium pressure mercury lamp was used as a light source. The light force was estimated with a UV light detector (Lutron UV-340), which in the range between 1.49–1.51 mW/cm². The temperature of the reaction mixture was kept around 25 °C by providing circulation of water throughout the experiments.

2.6. Evaluation of photocatalytic activity

The photodegradation of all the organic compounds was determined by measuring the change in absorption intensity as a function of irradiation time using the UV-vis spectroscopic analysis technique (PerkinElmer LAMBDA-35). The solution was irradiated and 5 mL sample was taken out at various time intervals and centrifuged at 6000-7000 rpm to eliminate the solid materials from the solution mixture and then analyzed spectrophotometrically at their characteristic wavelength. The concentration was calculated from the calibration curve obtained from the absorbance measured at various concentrations of the compound under investigation. For each experiment, the degradation rate of the model pollutant as a function of the irradiation time, i.e. first-order degradation kinetics, was determined from the initial slope obtained by a linear plot of the natural logarithm of the compound concentration.



Scheme S1

References

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