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Unexpected rapid photo-catalytic decolourisation/degradation of organic pollutants over highly active hetero junction based vanadium phosphate catalyst

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ABSTRACT

VPO materials are extensively used in the gas phase oxidation of *n*-butane to maleic anhydride. Herein we explored its catalytic activity towards decolourisation/degradation of organic pollutants. This investigation reports the remarkable photo catalytic activity of tungstate promoted vanadium phosphate (WO₃-VPO) toward photocatalytic decolourisation/degradation of pollutants. The catalyst i.e. 10 wt% WO₃-VPO showed unexpected efficacy i.e. 100% decolourisation of Rhodamine B (RhB) and 60% degradation of phenol under visible light irradiation with a short span of time i.e. 10 min. Several factors involving the significant results of the composite material than the neat sample have been studied in detail. The analysis of photoluminescence (PL) emission spectra, photoelectrochemical measurement and the cyclic voltammeter (CV) measurement of the composite materials greatly support the outstanding results of the catalyst. The remarkable efficiency of VPO materials in photocatalytic decolourisation/degradation would become another trademark in the photocatalysis society.

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1. Introduction

Environmental problems are normally recounted to waste and toxic organic with inorganic pollutants released in water bodies. The majority of the coloured and uncoloured effluents originated in contaminated waters contain organic dyes and phenolic compounds from textiles, dyestuff, dyeing industries, fertilizers and chemical production industries [1–3]. Several works on decolourisation/degradation of organic pollutants under visible light irradiation have been reported [4–11]. Our group and numerous researchers have evaluated the visible light driven photocatalytic decomposition of Rhodamine B (RhB) and phenolic compounds [12–17]. Very few literatures are reported on phosphate systems and WO₃ promoted phosphate catalysts for the oxidation of organic contaminants [18–22].

Herein, we report another phosphate system i.e. vanadium phosphate (VPO), a well known material which have extensively

been used industrially in the manufacture of maleic anhydride from *n*-butane [23]. However there is relatively little research on VPO materials in liquid phase reactions [24–31]. To tackle this tough issue, we put effort to explore the catalytic potential of VPO material towards some liquid phase reactions [24–26,29–31]. The results of our preceding works encouraged us to move into another catalytic world, photocatalysis. Photocatalytic materials have now gained much attention toward pollutant removal. In this concern, we put effort to explore the photocatalytic activity of WO₃-VPO catalysts for decolourisation/degradation of pollutants. The material has been well characterized and studied for the decolourisation of RhB and degradation of phenol. An outstanding result (100% decolourisation of RhB and 60% degradation of Phenol) has been achieved with a short span of time (10 min) with this system. Furthermore its reusability and stability makes it an efficient material for dye degradation.

2. Experimental section

2.1. Synthesis of vanadium phosphate (VPO)

The VPO precursor was prepared according to the procedure as follows V₂O₅ (5.0 g, SBMC, 98.5%) and o-H₃PO₄ (30 mL, 85%

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Aldrich) were refluxed in deionised water (120 mL) for 24 h. The yellow solid was recovered by vacuum filtration, washed with cold water (100 mL) and acetone (100 mL) and dried in air (110 °C, 24 h). Powder X-ray diffraction studies confirmed that the solid was the dihydrate, $\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$. The dihydrate (4 g) was refluxed with isobutanol (80 mL, 99%, Spectrochem) for 21 h, and the resulting hemi hydrate was recovered by filtration, dried in air (110 °C, 16 h), refluxed in deionised water (9 mL $\text{H}_2\text{O}/\text{solid}(\text{g})$) for 2 h, filtered hot, and dried in air (110 °C, 16 h).

2.1.1. Synthesis of tungstate promoted vanadium phosphate (WO_3 -VPO)

Different wt% WO_3 -promoted VPO catalysts were prepared by wetness impregnation method using water as solvent. Requisite amount of promoter (WO_3 as ammonium metatungstate) was dissolved in 30 mL of deionised water. Then desired amount of vanadium phosphate precursor in powder form was added. The resulting solution was vigorously stirred at room temperature till dryness. After impregnation; all materials were dried at 60 °C. The present promoted VPO final catalysts consist of 5, 10 and 15 wt% of WO_3 and are designated as $x\text{WO}_3$ -VPO ($x = 5, 10$ and 15). The study on different WO_3 loadings was carried out after thermal activation at 700 °C.

2.2. Preparation of working electrode

For the photo electrochemical study, Fluorine doped tin oxide (FTO) is taken as support because it does not show any photo-response in the solution. The working electrodes (as prepared materials-film electrodes) were prepared by electrophoretic deposition process. In this process, 30 mg of powder catalyst, 30 mL of acetone, and 20 mg of iodine powder were kept in 100 mL beaker followed by sonication for 10–15 min. Two parallel FTO coated electrodes were immersed in the solution with a 10–15 mm separation between them. Then 50A current and 60 V bias was applied to it for 0.03 min under potentiostat control to produce working electrode with coated area of $1 \text{ cm} \times 1.5 \text{ cm}$. The working electrodes are dried and used for electrochemical measurement.

2.3. Characterizations

The VPO catalysts were unambiguously characterized by XRD, UV-vis DRS, NH_3 -TPD, SEM, TEM, XPS, (these are explained elsewhere by the same authors [24]), PL, and Photocurrent measurement.

Powder X-ray diffraction was performed using an ENRAF Nonius FR590 X-ray generator with a $\text{Cu-K}\alpha$ source fitted with an Inel CPS 120 hemispherical detector. UV-vis investigation in diffuse reflectance mode was recorded in a UV-vis spectrophotometer (Varian, Australia). The spectrum was recorded in the range of 200–800 nm using boric acid as the reflectance standard. Surface morphology was observed via Scanning Electron Microscope (SEM, Hitachi S-3400N) by the help of gold sputtering. The transmission electron microscopy images were recorded using FEI, TECNAI G^2 20, TWIN and the images were recorded by using a Gatan CCD camera. The samples for electron microscopy were prepared by dispersing the powder in ethanol and dropping a very dilute suspension on carbon coated Cu grids. XPS measurements were made on a Kratos Axis Ultra DLD spectrometer using monochromatic $\text{Al-K}\alpha$ radiation. Samples were mounted using double-sided adhesive tape, and binding energy referenced to the C(1s) of adventitious carbon contamination is taken to be 284.7 eV. The acid character of the catalysts was studied using a TPD- NH_3 AutoChem 2920 (micromeritics, USA) chemisorption analyzer equipped with a thermal conductivity detector (TCD). About 0.1 g of the sample was housed in a quartz U-tube. Prior to analysis, the sample was

degassed at 110 °C for 2 h under N_2 flow (50 mL min^{-1}). It was then cooled down to 40 °C. The sample was saturated with NH_3 by the flow of 20% NH_3 -balanced He for 30–40 min (25 mL min^{-1}). The N_2 gas was flowed over the catalyst (50 mL min^{-1}) for 30 min to remove the physisorbed NH_3 . The temperature was increased from 40 to 800 °C with the flow of N_2 (50 mL min^{-1}) to get a TPD profile. The amount of NH_3 consumed was determined by the TCD detector, which gives the amount of acid sites in the sample. Before the experiment, the above equipment was calibrated using 20% NH_3 -balanced He to know the exact amount of gas consumed during the adsorption. Photoluminescence spectra were recorded with a LS 55 fluorescence spectrometer (Perkin Elmer) with excitation at 300 nm at room temperature. Current voltage was evaluated using a conventional pyrex electrochemical cell consisted of a prepared electrode as working electrode, a platinum wire and a Ag/AgCl electrode were used as counter and reference electrodes, respectively. The potential of the working electrode was measured by a potentiostat. The cell was filled up with an aqueous solution of 0.1 M Na_2SO_4 and the pH of the solution was kept at 6. Nitrogen gas was purged to deoxygenate the electrolyte in the cell which is applied for electrochemical measurements. The photoelectrochemical measurement was carried out using a potentiostat/galvanostat (Versastat 3, Princeton Applied Research) under illumination conditions ($\lambda \geq 400 \text{ nm}$). Irradiation fell on the conducting glass using a 300 W Xe lamp with a cold mirror and cut-off filters as necessary.

2.4. Photo-catalytic degradation studies

Concentrations of Rhodamine B (RhB) and phenol were resolved by evaluating the absorbance representative wavelengths with Varian Cary IE UV-vis spectrophotometer (Model EL 96043181). A standard solution of the dye and phenol was taken and the absorbance was analyzed at different wavelengths to attain a plot of absorbance versus wavelength. The wavelength resultant to maximum absorbance (λ_{max}) was calculated from this plot. Correspondingly, the λ_{max} for RhB and phenol were instituted to be 553 and 503 nm. Calibration curves were established between absorbance and concentration of the dye and phenol solution.

Decolourisation of RhB and degradation of phenol was studied using a batch technique under UV and visible light irradiation in an irradiation chamber (BS 02, Germany) at room temperature. 20 mL of 100 ppm of RhB and 0.03 g of catalyst was taken in a 100 mL stoppered conical flask in every experiment. The pH of the solution remains constant throughout the reaction i.e. 3.79 which was calculated by an Elico digital pH meter (Model LI-612) using a combined glass electrode (Model CL 51B). The solution was centrifuged by Research Centrifuge (Remi scientific works Mumbai) at 6200 rpm for 30 min and the clear residual dye concentration was determined by a Varian Cary IE UV-vis spectrophotometer (Model EL 96043181) fitted with Cary 100 software using 10-mm matched quartz cells. In the phenol analysis, the above stated condition was same but before spectrophotometric analysis, the color was developed by the addition of 2.5 mL of 0.5 M ammonium hydroxide solutions, followed by phosphate buffer to maintain the pH in the range of 7.7–7.9. After the pH adjustment, 1 mL of 4-aminoantipyrene and 1 mL of potassium ferricyanide were added to develop the red colour. Various parameters involving time, amount of catalyst have been studied in detail. The percentage of removal of RhB and phenol was calculated using the following formula:

$$\text{Percentageremoval}(\%) = (C_0 - C_t)/C_0 * 100$$

C_t is RhB and phenol concentration at time “t”, and C_0 is the initial concentration of the RhB and phenol under study.

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