



# Size Controlled Synthesis and Characterization of $V_2O_5/Al_2O_3$ Nanocomposites

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## ABSTRACT

$V_2O_5/Al_2O_3$  nanocomposites were synthesized by the wet chemical method, using mixed solvents such as water/ethanol and water/ethylene glycol (EG). The XRD results confirmed the crystalline nature and the presence of the orthorhombic structure of  $V_2O_5$  and hexagonal phase of  $\alpha-Al_2O_3$  nanocomposites. Transmission electron microscopy (TEM) analysis showed the agglomerated cluster nanoparticles in water/ethanol mediated  $V_2O_5/Al_2O_3$  nanocomposite; whereas well dispersed spherical nanoparticles with controlled particle size in water/ethylene glycol mediated  $V_2O_5/Al_2O_3$  nanocomposite. The Fourier transform infrared (FTIR) results confirmed the formation of a V–O and Al–O bond. Photoluminescence (PL) studies revealed that  $V_2O_5/Al_2O_3$  nanocomposites contain high intense peaks in the UV and visible region which are likely to have originated from the oxygen vacancies; and are potential material for electronic device applications and catalytic activities. The band gap energies of the samples prepared for water/ethanol and water/ethylene glycol obtained by the tauc plots are 3.6 and 3.62 eV respectively.

## 1. Introduction

Nanocomposite is a multiphase solid material in which one of the phase exhibit nano-dimensions. Introducing another material to synthesize composite material is an effective way to improve the physical and chemical properties due to its multi phases [1]. The unique physical, chemical and mechanical properties including a very high surface area and easy surface modifications allow preparation of nanocomposite materials with novel properties and characteristics [2]. Specifically, many researchers reported the composite photocatalyst which increases the charge separation and hence increases the photocatalytic efficiency; as well as the nanocomposites can reduce the band gap, extending the absorbance range to visible region leading to electron-hole pair separation under irradiation and consequently, achieving a higher photocatalytic activity [3–8]. In addition, the nanocomposites with different electronic and chemical properties represent a promising direction to enhance the sensor selectivity and sensitivity [9–11].

Vanadium pentoxide (Vanadia- $V_2O_5$ ,  $E_g = \sim 2.2\text{--}2.8\text{ eV}$ ) is one of the most attractive semiconductor material has greatly attracted due to its potential applications such as catalysis, gas sensors, optoelectronic devices, electrochemistry and lithium ion batteries [12–14]. In particular, vanadium oxide based composites have received much attention because of their unique physico-chemical properties [14–16]. Aluminum oxide (Alumina- $Al_2O_3$ ,  $E_g = 8.8\text{ eV}$ ) is one of the most

important ceramic material and it used for wide range of applications such as electronics, optoelectronics, thermoluminescent dosimeters, lasers, light emitting display, cutting tools, spark plugs, solar cells, gas sensing, catalysis, bio medical areas and in modern surgery due to its excellent properties such as high strength at elevated temperatures, hardness, high melting point, thermal conductivity, chemical inertness, large band-gap, abrasion resistance and thermal shock resistance, mechanical properties with good chemical stability and solubility [17–20]. Particularly,  $V_2O_5/Al_2O_3$  nanocomposite have attracted many researchers due to its prominent catalytic activity, sensing behavior, and water treatment capacity with better oxygen storage capacity and thermal stability have been considered as an outstanding material for potential applications [21–23].

The enhanced optical and electrical properties of nanocomposites are newly emerging areas of potential applications. In recent years great efforts have been made in developing an easy process to prepare well dispersed nanoparticles with controlled particle size is the great importance because the optical, electrical and magnetic properties of these nanoparticles depend strongly on their size [24–26]. Various research groups reported, using different solvents, which strongly influenced the size of the nanoparticles [27,28]; In particular, using mixed solvents which may play an important role in synthesizing size and morphology controlled nanomaterials [29–31]. In addition, many researchers focused to synthesis of nanomaterials with enhanced

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photoluminescence property due to its various applications in display devices, cathode ray tubes, fluorescence lamps, color plasma display panels, LEDs and other opto-electronic devices. In our work shows the synthesis of  $V_2O_5/Al_2O_3$  nanocomposites by sol-gel method using different mixed solvents such as water/ethanol and water/ethylene glycol and investigate the structural, optical, and photoluminescence properties. There are few reports on the synthesis of  $V_2O_5/Al_2O_3$  nanocomposites [21–23,32–38]; however, till date there is no report in the literature to the synthesis of the size controlled  $V_2O_5/Al_2O_3$  nanocomposites and its optical and room temperature photoluminescence studies.

## 2. Experimental Section

### 2.1. Materials

The aluminum nitrate nonahydrate ( $Al(NO_3)_3 \cdot 9H_2O$ ), ammonium meta vanadate ( $NH_4VO_3$ ), ammonium hydroxide ( $NH_4OH$ ), ethanol ( $C_2H_5OH$ ) and ethylene glycol ( $HO-CH_2-CH_2-OH$ ) and distilled water were purchased from Aldrich. All the chemicals used in this work were analytical graded and used without further purification.

### 2.2. Synthesis of $V_2O_5/Al_2O_3$ Nanocomposites

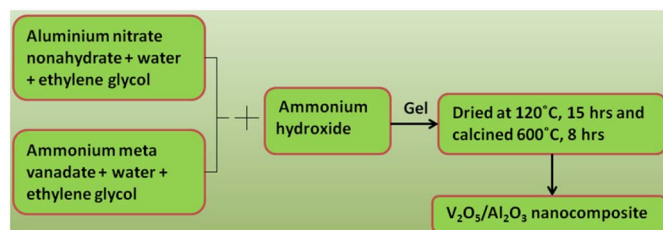
In a typical synthesis, 0.1 M of  $Al(NO_3)_3 \cdot 9H_2O$  and 0.1 M of  $NH_4VO_3$  solutions were prepared separately in mixed solvents of water/ethanol (1:1) and stirred for 15 min. The above solutions were mixed together under constant stirring and to the above solution an aqueous ammonium hydroxide ( $NH_4OH$ ) solution was added drop wise in order to attain the pH value of 6.5. The stirring was continued for 2 h at room temperature and the resulting gel product was dried for 15 h at 120 °C. After that it was transferred into the alumina crucible and calcined in a muffle furnace at 600 °C for 8 h. The same procedure was adopted for other mixed solvent water/ethylene glycol mediated  $V_2O_5/Al_2O_3$  powder to be synthesized. Synthesis of water/ethylene glycol assisted  $V_2O_5/Al_2O_3$  nanocomposite is as shown in Scheme 1.

### 2.3. Characterization Details

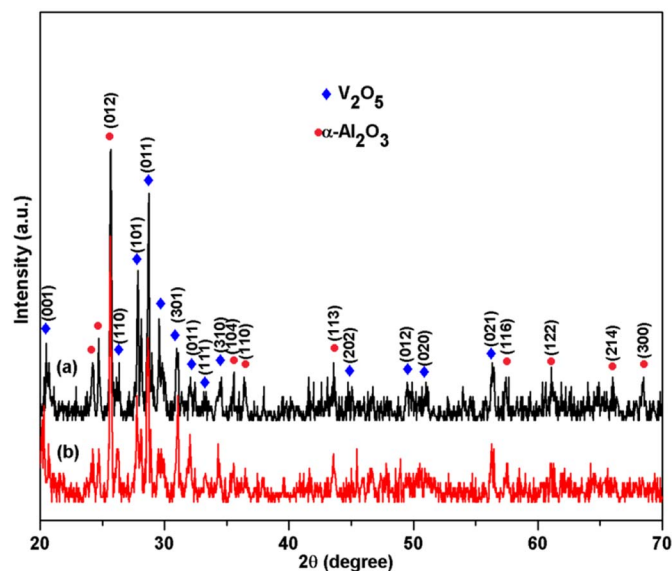
The samples were analyzed by using a powder X-ray diffractometer (Schimadzu model: XRD 6000 using  $CuK\alpha$  ( $\lambda = 1.5417 \text{ \AA}$ ) radiation. The Fourier transform infrared (FTIR) spectra were taken by a Bruker IFS 66 V FTIR spectrometer. The EDX studies were carried out by the Philips model CM 20. High-resolution images and selected area electron diffraction (SAED) patterns were observed with a JEOL JEM-2200FS transmission electron microscope (TEM) operating at 200 kV. The UV–vis absorption studies of the as synthesized samples were recorded using the Varian Cary 5E UV–vis Spectrophotometer. The photoluminescence emission spectra were carried out on a Fluoromax-4 spectrofluorometer with a Xe lamp as the excitation light source.

## 3. Results and Discussion

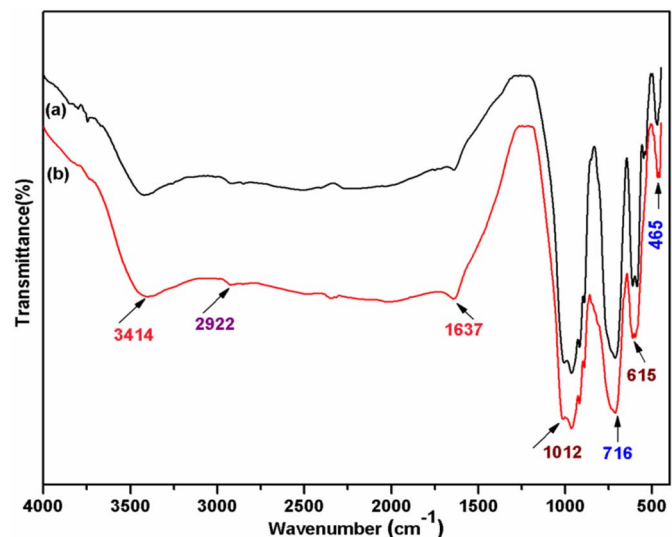
Fig. 1(a, b) shows the XRD patterns of the  $V_2O_5/Al_2O_3$



**Scheme 1.** The schematic diagram of the formation of water/ethylene glycol mediated  $V_2O_5/Al_2O_3$  nanocomposite.



**Fig. 1.** Powder X-ray diffraction patterns mixed solvents mediated  $V_2O_5/Al_2O_3$  nanocomposite: (a) water/ethanol, and (b) water/ethylene glycol.



**Fig. 2.** FTIR spectra of mixed solvents mediated  $V_2O_5/Al_2O_3$  nanocomposite: (a) water/ethanol, and (b) water/ethylene glycol.

nanocomposite prepared in water/ethanol and water/ethylene glycol. The diffractogram confirms the formation of orthorhombic structure of  $V_2O_5$  (JCPDS No: 75-457) and (JCPDS file no: 89-0612) with its diffracted peaks at  $2\theta$  values of 20.5°, 26.3°, 27.9°, 28.7°, 31.0°, 32.1°, 33.2°, 34.5°, 44.8°, 49.5°, 51.0°, and 56.3° which correspond to (001), (110), (101), (011), (301), (011), (111), (310), (202), (012), (002), and (021) crystal planes respectively [39,40]. Similarly, hexagonal phase of  $\alpha-Al_2O_3$  (JCPDF 75-1862 and JCPDS # 46-1212) was confirmed with its diffraction peaks at  $2\theta$  values of 25.6°, 35.4°, 36.5°, 43.5°, 57.5°, 61.09°, 66.0° and 68.5° which corresponds to (012), (104), (110), (113), (116), (122), (214) and (300) crystal planes respectively [17,41,42]. The extra diffraction peaks observed in patterns at  $\sim 24^\circ$  and  $\sim 29.5^\circ$  is belonging to alpha alumina and vanadia phase [40,42]. The average crystallite size of the  $V_2O_5/Al_2O_3$  nanocomposite at different mixed solvents was found by the Debye-Scherrer equation (Eq. (1)) from full width at half maximum (FWHM) values of the  $V_2O_5$  and  $Al_2O_3$  planes.

$$D = 0.89\lambda / \beta \cos \theta \quad (1)$$

where  $\lambda$  represents the wavelength of the X-ray,  $\theta$  indicates Bragg's

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