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Simple synthesis of new nano-sized pore structure vanadium pantoxide (V_2O_5)

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ABSTRACT: New forms of vanadium oxide Nanoporous were fabricated using a simple chemical synthesis method. Vanadium pentoxide (V_2O_5) Nanoporous were synthesized by sodium metavanadate as precursor and ethylene glycol as surfactant. The samples were characterized by high resolution transmission electron microscopy (HRTEM), field effect scanning electron microscopy (FESEM) and X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and UV-Vis spectrophotometer. Structures of the nanoparticles were characterized by XRD technique to identify α-vanadium and γ-vanadium. The smallest particle size of sponge-like as-prepared sample was around 20 nm in diameter and for annealed sample was around 25 nm as estimated by XRD technique and direct HRTEM observation. The surface morphological studies from SEM depicted increasing dimension of sponge-like shaped structure by increasing ratio of the EG surfactant from 25 nm to 30 nm. The sharp peaks in FTIR spectrum determined the purity of V_2O_5 nanoparticles.

Keywords: *Ethylene glycol (EG); Nano-porous; Surfactant; Synthesis; Vanadium pentoxide*

INTRODUCTION

Vanadium oxide a strongly correlated oxide with its first-order metal-insulator transition a little above room temperature, has been well-studied over the last fifty years. Divanadium pentoxide (V_2O_5), the most stable form in the V-O system, has been at the front position of applied research due to its unique physio-chemical properties. The layered crystal structure of vanadium oxide nanos-tructures are currently drawn attention for the application of super capacitors and chemical sensors (Reddy, *et al.*, 2006, Liu, *et al.*, 2005), electrical and optical properties, has led to wide potential applications including rechargeable lithium batteries (Lampe-Onnerud, *et al.*, 1995) and optical data storage media (Balberb, *et al.*, 1975, Cam, *et al.*, 2006). Vanadium pentoxide exhibits a number of polymorphs, including α-V_2O_5 (orthorhombic) (Singh, *et al.*, 2008), β-V_2O_5 (monoclinic or tetragonal) and γ-V_2O_5 (orthorhombic) (Su, *et al.*, 2009). The α-V_2O_5 phase is the most stable phase and the other two phases can be converted from the α-V_2O_5 phase under high temperature and high pressure (Balog, *et al.*, 2007). Vanadium oxide based catalysts are widely used in a variety of chemical reactions like reduction of NOx or partial oxidation of alkanes (Yang, *et al.*, 2008). Synthesis of a wide range of nanostructures predominantly, high or-

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der nanomaterials with well-defined geometries such as nanorods, nanobelts, nanotubes and nanowires have attracted fabulous interest due to their novel chemical and physical properties and their prospective applications in fabricating electronic, magnetic, optical, electrochemical devices (Mai, *et al.*, 2008, Lions, *et al.*, 2009). Vanadium in V_2O_3 is formally +3 with two 3d electrons per V atom. There is another phase transition in V_2O_3 at about 520K, above which the conductivity is again lower than in the metallic phase. VO has the sodium chloride crystal structure and vanadium is formally +2. Bulk vanadium monoxide has many intriguing properties that are closely related to the issue of stoichiometry (Mousavi, *et al.*, 2013, Vijaykumar, *et al.*, 2012, Dinh, *et al.*, 2010). Comparing with the other microstructure, nano-porous structure may be the most controllable, and its structure could significantly improve the battery life and increase Li+ diffusion rate and optical data storage media. In this paper, novel vanadium oxide nanoparticles are fabricated by using sol-gel method. Structural, optical and surface morphological properties are discussed by XRD, HR-TEM, FESEM and FTIR analyses.

EXPERIMENTAL DETAIL

Vanadium oxide nanoparticles were synthesized by a simple synthesis according to the following manner. Firstly, 0.3 g sodium metavanadate was completely dissolved in 50 mL pure water with stirring at room temperature. Ammonium chloride (0.5 g) was then added to the solution until dissolve completely. After 5 min, 2 mL ethylene glycol (EG) was added to the solution as complex agent and synthesis temperature was increased to 85°C. The color of solution changed from bright color to yellow color by adding EG. The Ph was fixed to 2 by adding sulfuric acid (1mL) drop by drop to the solution and the color changed to the orange yellow color. After one hour, during the process, the color of solution changed to obscure color. The product were evaporated for 2 hours, cooled to room temperature and finally calcined at 600°C for 4 hours. All analyses were done for samples without any washing and more purification. The specification of the size, structure and surface morphological properties of the as-synthesis and annealed vanadium oxide nanoparticles were carried out. X-ray diffractometer (XRD) was used to identify the crystalline phase and to estimate the crystalline size. The XRD pattern were recorded with 2θ in the range of 4-85° with type X-Pert Pro MPD, Cu-Kα: λ = 1.54 Å. The morphology was characterized by field emission scanning electron microscopy (SEM) with type KYKY-EM3200, 25 kV and transmission electron microscopy (TEM) with type Zeiss EM-900, 80 kV. All the measurements were carried out at room temperature.

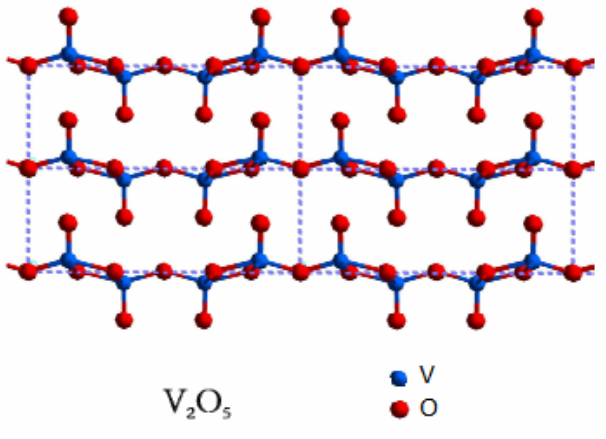

Fig. 1: Schematic structure of V_2O_5.

RESULTS AND DISCUSSION

X-ray diffraction (XRD) at 40Kv was used to identify crystalline phases and to estimate the crystalline sizes. Fig. 2(a) shows the XRD pattern of vanadium oxide before annealing. Fig. 2(b) shows the X-ray diffraction patterns of the powder after heat treatment at 600°C for 4 hours. The XRD patterns showed this sample have sharp peaks 2θ angle at the peak position at 25.2°, 32°, 33.5°, 37.5°, 45° and 51° with (101), (400), (011) , (301), (411) and (002) diffraction planes, respectively are in accordance rhombohedral structure of the V_2O_5 phase. The mean size of the ordered α-V_2O_5 nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Sherrer formula according to equation the following:

$$D = \frac{0.89\lambda}{B\cos\theta} \quad (1)$$

where, 0.89 is the shape factor, λ is the X-ray wave-

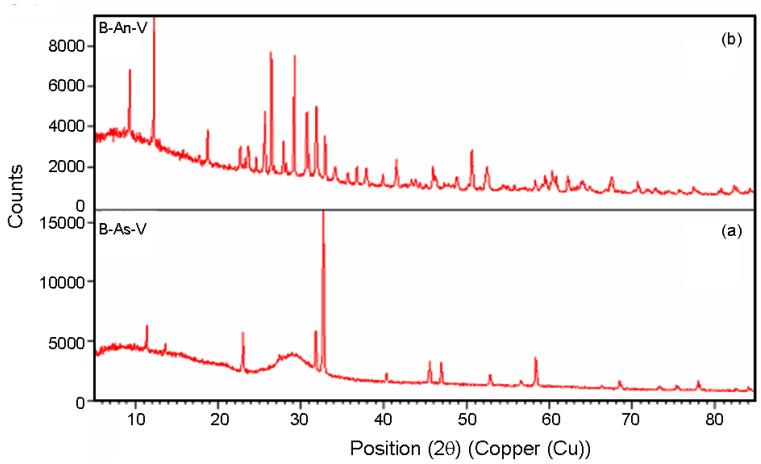

Fig. 2: XRD pattern of vanadium pentoxide (a) as-synthesixed and (b) at 600°C.

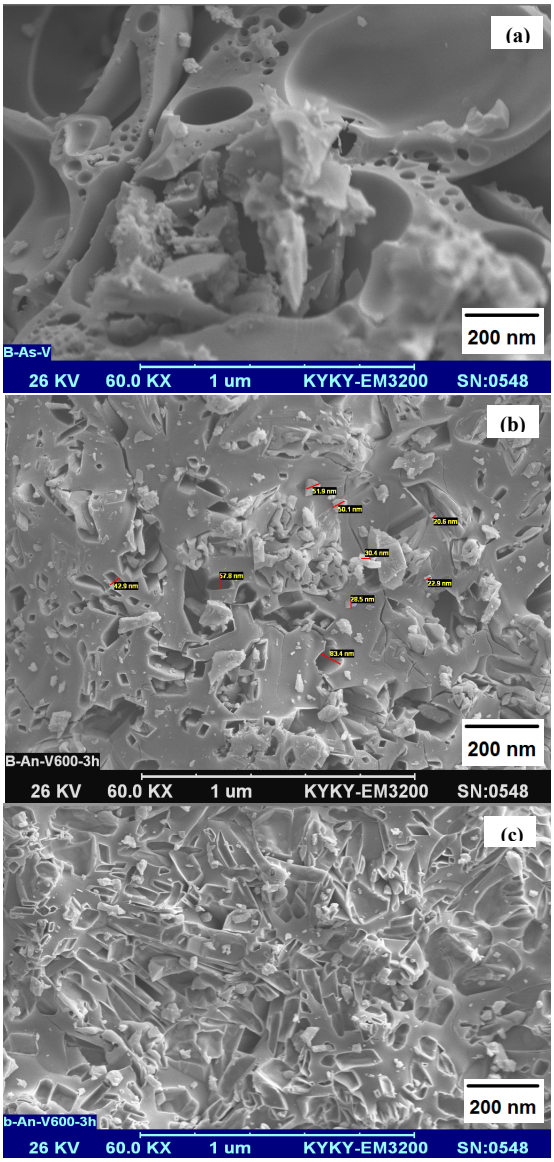

Fig. 3: SEM images of the V_2O_5 nanoporus (a) as-prepared sample (b) annealed at 600°C with 2 mL EG and (c) annealed at 600°C with 4 mL EG.

length, B is the line broadening at half the maximum intensity (FWHM) in radians, and θ is the Bragg angle. The mean size annealed V_2O_5 nanoparticles was around 20 nm from this Debye-Sherrer equation.

SEM analysis was used for the morphological study of nanoparticles of V_2O_5 samples. These analyses show high porosity structure emerged in the samples surface by increasing annealing temperature. With increasing temperature the morphology of the particles changes to the sponge-like shaped. Fig. 3(a) shows the SEM image of the as-prepared V_2O_5 nanoparticles with formation of clusters. Fig. 3(b) shows the SEM image of the annealed sponge-like shaped nanoporus V_2O_5 at 600°C for 4 hours. The smallest dimension of V_2O_5 nanoporous formed was about 25 nm. The effect of EG surfactant on the size of nanoporus were investigated and the results showed that with increasing the ratio of the surfactant from 2 mL (Fig. 3b) to 4 mL (Fig. 3c) the dimension of nanoporus increase from

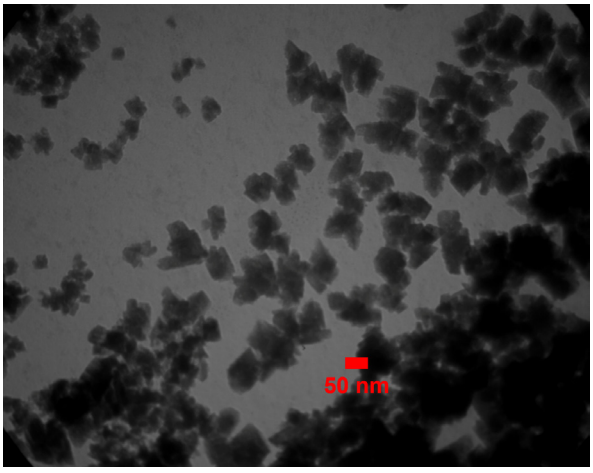

Fig. 4: TEM images of the as-prepared V_2O_5 nanoporus.

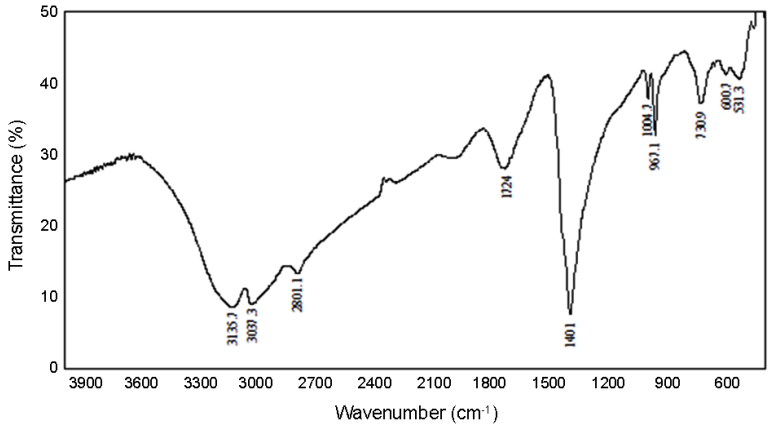

Fig. 5: FTIR spectrum of V_2O_5 sample.

25 to 30 nm. TEM analysis was carried out to confirm the actual size of the particles, their growth pattern and the distribution of the crystallites. Fig. 4 shows the as-synthesized TEM image of as-synthesized V_2O_5 particles prepared by chemical route. The vanadium oxide nanoparticles were formed with size of about 20 nm for smallest one.

According to Fig. 5, the infrared spectrum (FTIR) of the synthesized V_2O_5 nanoparticles was in the range of 400-4000 cm^{-1} wavenumber which identify the chemical bonds as well as functional groups in the compound. The large broad band at 3135 cm^{-1} and 3037 are ascribed to the O-H and C-H groups. The absorption picks around 1401 cm^{-1} is due to the bending vibration of C=C vibration. FTIR spectra of V_2O_5 nanoparticles exhibited three characteristic vibration modes: V=O vibrations at 967 cm^{-1}, the V-O-V symmetric stretch around 531 cm^{-1} and the V-O-V asymmetric stretch at 730 cm^{-1}. As clearly seen, the bands appearing, between 950 and 1020 cm^{-1} were assigned to a vanadly stretching modes (δ V-O). Bands between 700 and 900 cm^{-1} were ascribed to the bridging V-O-V stretching.

CONCLUSIONS

Novel nano-porous vanadium pentoxide particles were successfully prepared using sodium metavanadate as precursor and ethylene glycol as surfactant. XRD spectrum shows rhombohedral (hexagonal) structure of α-V_2O_5 annealed at 1000°C. From SEM images, it is clear that with increasing temperature the morphology of the particles changes to sponge-like shaped. The effect of EG surfactant on the size of nanoporus were studied and the results showed that with increasing the ratio of the surfactant from 2 mL to 4 mL the dimension of nanoporus increase from 25 to 30 nm. TEM image exhibits that the uniform as-synthesized V_2O_5 nanoporous prepared by chemical route with an average diameter about 20 nm. From the FTIR data, it is shown the presence of V-O and V-O-V stretching mode of V_2O_5.

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