

# Chemical synthesis of vanadium oxide ( $V_2O_5$ ) nanoparticles prepared by sodium metavanadate

## Abstract

Vanadium oxides ( $V_2O_5$ ) nanoparticles have been prepared using a simple chemical method by sodium metavanadate as precursor and Cetyl trimethylammonium bromide (CTAB) as surfactant. The samples were characterized by high resolution transmission electron microscopy (HRTEM), field effect scanning electron microscopy (FESEM) and X-ray diffraction (XRD). As there are many forms of vanadium oxides produced during this process, x-ray diffraction (XRD) technique was used to identify  $V_2O_5$  phases. The size of as-prepared nanoparticles was around 5 nm and average diameter of annealed one was around 10 nm. The effect of CTAB surfactant on the particle morphology showed that the size of particles reduce to 10 nm in presence of CTAB surfactant. FTIR spectrum showed the presence of V-O and V-O-V stretching mode.

**Keywords:**  $V_2O_5$ , Nanocrystals, CTAB, Chemical synthesis, Surfactant

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**Abbreviations:** EC, Electro Chromic; CTAB, Cetyl Trimethyl Ammonium Bromide; TEM, Transmission Electron Microscopy; XRD, X-ray Diffractometer; SEM, Scanning Electron Microscopy

## Introduction

Nano-materials have unique physical properties that have attracted more and more attention as a cathode in rechargeable ion batteries and selective gas sensors such as ammonia because of their high surface area and redox activity.<sup>1-3</sup> Biological activity of vanadium pentoxide nanomaterial depends on factors such as the type of the derivative, manner of its administration, dose, length of treatment, and also individual- and species-specific sensitivity to the administered compound.<sup>4</sup>  $V_2O_5$  nanomaterial is amphoteric in nature. Vanadium is correlated to its degree of oxidation (vanadyl\vanadate ion) and chemical form (organic\inorganic ligand).<sup>5-7</sup> The existence of the various vanadate species depends on the pH and on the total concentration of vanadium. Their occurrence can be accounted for condensation equilibrium; it is evident that only in very dilute solutions are monomeric vanadium ions found, and increases in concentration, particularly if the solution is acidic, lead to polymerization.<sup>8-10</sup> Vanadium oxygen systems ( $V_2O_5$ ,  $VO_2$ ) are prototype strongly-correlated materials that have been widely-studied by theoretical and experimental condensed-matter and materials community for more than half a century.<sup>11</sup> Vanadium oxide is a well-known catalyst among various metal oxides, and so many fundamental studies have been developed wide-spreadingly centering on catalytic oxidation.<sup>12</sup> They show metal-semiconductor transition, which implies an abrupt change in optical and electrical properties.<sup>13</sup> That is why this oxide is used in thermal sensing and switching. Vanadium pentoxide based materials are known to display several types of chromogenic effects, as a window for solar cells and for transmittance modulation in smart windows with potential applications in architecture, automotives and nanomedicine.<sup>14</sup> It shows an atypical behaviour because it cannot be defined exactly either as a cathodically or as anodically colouring material.  $V_2O_5$  exhibit multi-colored electrochromism allowing the use in electrochromic (EC) displays color filters and other optical devices.<sup>15</sup>

Transition metal oxides have been a subject of research in recent years in view of their fundamental and technological aspects.

Among these, vanadium creates many compounds with oxygen; these have different structural, optical and chemical properties. Meaningful differences between the properties of different phases of vanadium oxides like  $VO$ ,  $VO_2$ ,  $V_2O_3$  and  $V_2O_5$  depend on their structure, which determines other properties.<sup>16,17</sup> Different forms of vanadium oxides can be obtained by changing the deposition process parameters, or by post-process treatment, e.g., additional annealing.<sup>18</sup> From the application point of view, the most interesting vanadium oxides are  $VO_2$  and  $V_2O_5$ . Vanadium dioxide is a very good candidate for thermo chromic coatings due to the change of properties from semiconducting to semimetal at 68°C. Vanadium pentoxide ( $V_2O_5$ ) is a thermodynamically stable form which exhibits electrochromic properties.  $V_2O_5$  thin films can also be used in optical filters, reflectance mirrors, smart windows and surfaces with tunable emittance for temperature control of space vehicles [19]. It can be received by selecting deposition parameters or by the annealing of  $VO_2$  above 350°C [20]. In this article, vanadium oxide nanoparticles are fabricated by using sol-gel method. Structural and surface morphological properties have been studied.

## Experimental detail

The samples were synthesized by chemical synthesis according to the following manner. At First, 0.1g sodium metavanadate was completely dissolved in 100 mL pure water with stirring at room temperature. Ammonium chloride (1.2g) was then added to the solution until dissolve completely. The color of solution changed from muddy color to transparent color which after a few minutes changed to fuggy color. After 10 minutes, 0.5g, Cetyl trimethylammonium bromide (CTAB) was added to the solution and synthesis temperature was increased to 80oC. The color of solution changed from orange color to dark brown color. The pH was between 6 and 8 during the synthesis. After one hour, the color of solution changed to transparent yellow color. The product were evaporated for 2 hours, cooled to room temperature and finally calcined at 600oC for 4 hours. All analyses were done for samples without any washing and more purification. The specification of the size, structure and optical 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  $2\theta$  in the range of 4-85o with type X-Pert Pro MPD, Cu-K $\alpha$ :  $\lambda =$

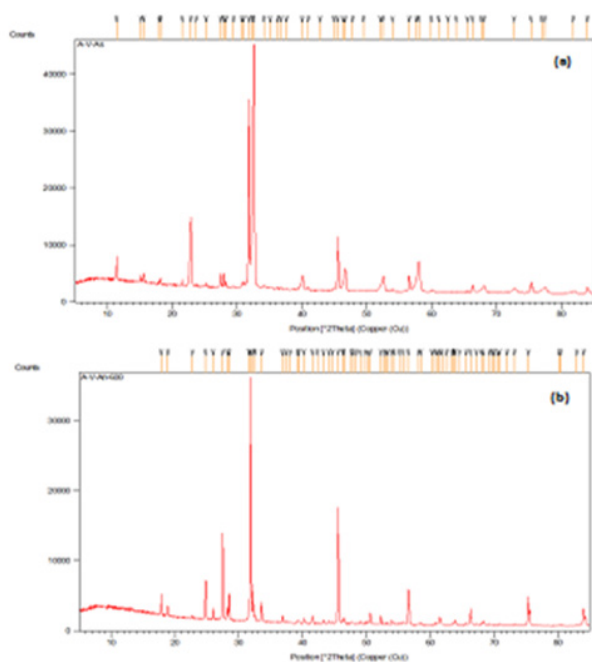
1.54 Å. The morphology was characterized by field emission scanning electron microscopy (SEM) with type KYKY-EM3200, 25 kV and transmission electron microscopy (TEM) with type Zeiss EM-900, 80 kV. All the measurements were carried out at room temperature.

## Result and discussion

Figure 1a shows the XRD pattern of aluminium oxide before annealing. Figure 1b 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 with (101), (400), (011), (301), (411) and (002) diffraction planes, are in accordance rhombohedral structure of the V<sub>2</sub>O<sub>5</sub> phase. The mean size of the ordered V<sub>2</sub>O<sub>5</sub> nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Sherrer formula according to equation the following:

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

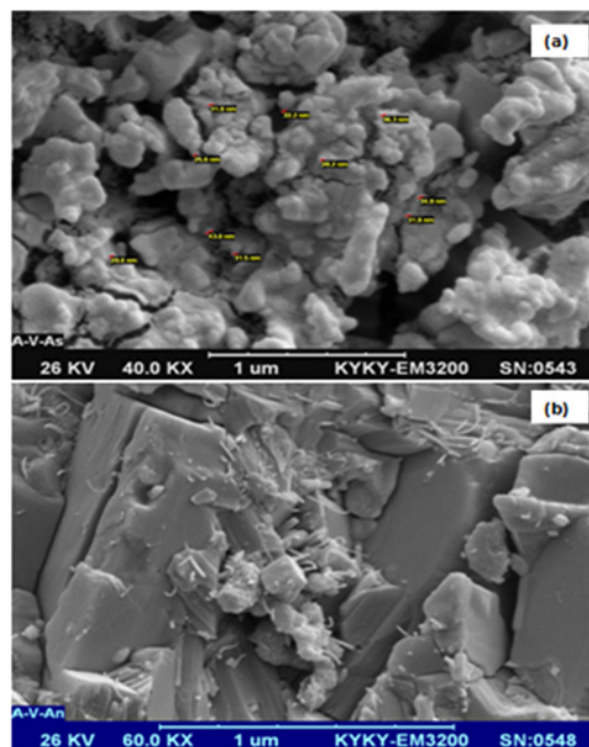
where, 0.89 is the shape factor,  $\lambda$  is the x-ray wavelength, B is the line broadening at half the maximum intensity (FWHM) in radians, and  $\theta$  is the Bragg angle. The mean size annealed V<sub>2</sub>O<sub>5</sub> nanoparticles was around 10 nm from this Debye-Sherrer equation.



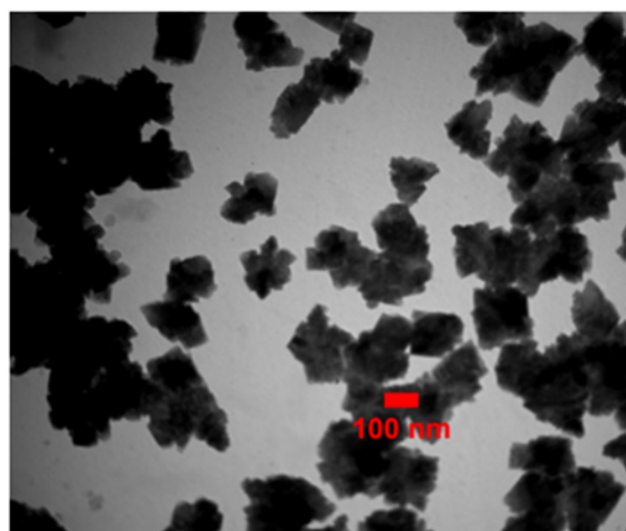
**Figure 1** XRD analyses of (a) as-synthesized (b) annealed V<sub>2</sub>O<sub>5</sub> nanoparticles at 600 °C.

In the next step, SEM analysis was used for the morphological study of nanoparticles of V<sub>2</sub>O<sub>5</sub> samples. These analyses show the nanoparticles are appeared in the samples by increasing annealing temperature. Figure 2a shows the SEM image of the as-prepared V<sub>2</sub>O<sub>5</sub> grains with formation of clusters. Figure 2b shows the SEM image of the annealed V<sub>2</sub>O<sub>5</sub> nanoparticle at 600°C for 4 hours. The smallest diameter of V<sub>2</sub>O<sub>5</sub> nanoparticles formed was about 10 nm.

TEM analysis was carried out to confirm the actual size of the particles, their growth pattern and the distribution of the crystallites. Figure 3 shows the as-synthesized TEM image of V<sub>2</sub>O<sub>5</sub> nanoparticles prepared by sol-gel route. The uniform structure of the vanadium oxide was formed in the range size of 5-10 nm.



**Figure 2** SEM images of the (a) as-prepared V<sub>2</sub>O<sub>5</sub> (b) annealed one at 600°C.



**Figure 3** TEM image of the as-prepared V<sub>2</sub>O<sub>5</sub> sample.

FTIR spectra of the samples were analyzed in the range of 400-4000 cm<sup>-1</sup> wave number which identifies the chemical bonds as well as functional groups in the compound. The large broad band at 3135 cm<sup>-1</sup> and 3037 are ascribed to the O-H and C-H groups. The absorption picks around 1401 cm<sup>-1</sup> is due to the bending vibration of C=C vibration. FTIR spectra of V<sub>2</sub>O<sub>5</sub> nanoparticles exhibited three characteristic vibration modes: V=O vibrations at 967 cm<sup>-1</sup>, the V-O-V symmetric stretch around 531 cm<sup>-1</sup> and the V-O-V asymmetric stretch at 730 cm<sup>-1</sup> (Figure 4). As clearly seen, the bands appearing, between 950 and 1020 cm<sup>-1</sup> were assigned to a vanadyl stretching modes ( $\delta$  V-O). Bands between 700 and 900 cm<sup>-1</sup> were ascribed to the bridging V-O-V stretching.

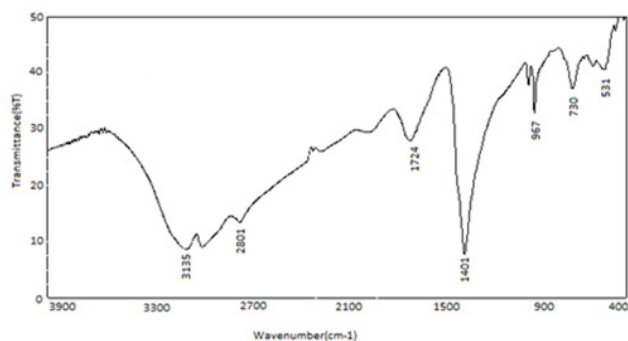


Figure 4 FTIR spectrum of as-grown  $V_2O_5$  sample.

## Conclusion

Vanadium oxide nanoparticles were successfully prepared using simple chemical method by sodium metavanadate as precursor and CTAB as surfactant. XRD spectrum shows rhombohedral structure of  $V_2O_5$  annealed at 600°C. From SEM images, it is clear that with increasing temperature the morphology of the particles changes to nanoparticle shaped and the size of particles decreases to 10 nm. TEM image exhibits that the uniform as-synthesized  $V_2O_5$  nanoparticles in the range size of 5-10 nm. Finally FTIR spectrum shows the presence of V-O and V-O-V stretching mode of  $V_2O_5$ .

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## Conflicts of interest

None.

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