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Citric Acid Assisted Facile Synthesis Of Single Phased Crystalline Micro Vanadium Pentoxide Powder

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ABSTRACT

The increased interest in the synthesis of V_2O_5 micro/nanostructures is due to their potential applications in catalysis and in optical and electrochemical devices. Citric acid assisted facile synthesis of highly pure micro vanadium pentoxide powder was done using ammonium metavanadate as the precursor. The method explores the advantage of simple solution mixing of the precursor and the modifier solutions at room temperature followed by thermal decomposition at 400 °C. XRD analysis of the prepared sample established the formation of phase pure V_2O_5 with an orthorhombic structure. FTIR spectra confirmed the chemical composition of the formed V_2O_5 material. FESEM images at high magnification revealed that the microcrystals with different shapes were formed by the agglomeration of V_2O_5 nanoparticles which was confirmed by TEM.

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INTRODUCTION

Vanadium oxide nanomaterials and their derivatives attract the attention of researchers because of their distinctive physico-chemical properties and potential applications in numerous areas (Shahid, M. *et al*., 2010). Because of the rapid increase in the resistivity of vanadium oxides, vanadium pentoxide in particular, they are largely used in the fields of solid state ionics, microelectronics and optoelectronics (Shin, D.H., *et al*., 2006). Nano-sized vanadium pentoxides of different morphology are used in high-energy lithium batteries to improve the capacity, voltage (versus the anode material), reversibility, and stability in chemical sensorsand photocatalysis (Li, B., *et al*., 2006). V_2O_5nanomaterials are also used as catalysts for various reactions, as cathode for lithium batteries and as electric field-effect transistors (Zhang, G., *et al*., 1997). Since V_2O_5 shows multi coloured electro-chromism, they are applied in different optical devices like colour filters, Electrochromic display (ECD) (Lao, Z.J. *et al*., 2006) and smart windows (Nagase, K., *et al*., 1992). The application of vanadium pentoxide electrode for electrochemical capacitors (EC) is because of its capability to exist in different oxidation states and low cost (Lao, Z.J. *et al*., 2006). The partially filled d-orbitals of vanadium are reasonable for the existence of different oxidation states which is responsible for magnetic, catalytic and electronic properties of V_2O_5 (Asim, N., *et al*., 2009). Different physical and chemical methods have been tried to prepare V_2O_5 with desired size and morphology. Wang *et al*. (2011) and Shevchuck *et al*. (2011) reported that monodisperisty and the size of V_2O_5nanomaterials are important factors that to be concerned about their application in technological and industrial fields. Commonly dry methods like sputtering and vacuum evaporation are adopted for the synthesis of V_2O_5 powder, but for the production of large scale films, wet routes like as sol-gel and electrodeposition are found to be more suitable. Synthesis of vanadium oxide nanotubes and gels is a really interesting area because of their improved ionic and electronic properties. In addition, many reports are there on the synthesis of V_2O_5nanoparticles by various methods like hydrothermal, soft template, pulsed laser ablation (Asim, N., *et al*., 2009; Liu, J., *et al*., 2006; Levi, R. *et al*., 2010). Self-assembling $(NH_4)_{0.5}V_2O_5$ nanowires are prepared by hydrothermal treatment of ammonium metavanadate with water[12]. By using V_2O_5 sols as precursor and hexadecylamine (HDA) as structure-directing template, Chen *et al*. (2004) prepared vanadium oxide nanotubes (VO_x-NTs) by a modified sol-gel method followed by hydrothermal treatment. Hydrothermal treatment of NH_4VO_3 in the presence of polymer polyethylene glycol 4000 resulted in the formation of single crystalline vanadium oxide nanobelts (Phetmung, H., *et al*., 2008).

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There are a number of methods have been developed for the synthesis of micro/nanostructures, but they require high molecular weight surfactants or special instruments (Shahid, M. *et al*., 2010). So the development of low cost and simple procedure for the synthesis of micro/nano V_2O_5 material is challenging. In our work, we provide a low cost, high yielding and facile method for the synthesis of phase pure crystalline micro V_2O_5 powder with different shapes consisting agglomerated V_2O_5nanospheres. We made use of citric acid as the structure modifier since it is non-toxic, less expensive and easily dissolvable in the aqueous reaction medium (Chen, Y., *et al*., 2011).

METHODS AND MATERIALS

Preparation of the sample:

Ammonium metavanadate and citric acid were purchased from Alfa Aesar and Fisher Chemicals respectively. 1.83g citric acid ($C_3H_8O_7$) was stirred in 30 ml distilled water for 2 minutes. 1.6 g ammonium metavanadate (NH_4VO_3) was added into it under stirring. The whole mixture was then stirred continuously for 8 h. It was then dried overnight at 80°C and was calcined at 400°C.

Characterisation of the V_2O5 samples:

Powder X-ray diffraction analysis was performed using Bruker D8 Advance powder diffractometer with Cu Kα radiation of wavelength 0.15406 nm. The samples were scanned from 3 to 80° in 0.025° step rise. The mean crystalline size for each sample was calculated using the Scherrer equation. The surface morphology of the sample was determined by field emission scanning electron microscopy (FESEM) using Carl Zeiss Evo Ma10 apparatus. The scanning electron micrographs were obtained at 10 kV. Fourier Transform-Infrared pattern was recorded on a thermo scientific NICOLET 6700 apparatus in transmission mode in the region 400-4000 cm-1. Transmission electron microscopy (TEM) images were taken by Philips CM-12 operated at a voltage of 100 kV.

RESULTS AND DISCUSSION

The XRD pattern of the prepared sample is shown in figure 1. All characteristic peaks can be indexed to the orthorhombic phase of V_2O_5 (space group: Pmmn, a= 11.512 Å, b= 3.564 Å, c= 4.368 Å) which agrees with reported data according to JCPDS No. 41-1426 24. No peaks of impurities were detected for the sample. Sharp and strong peaks in the XRD pattern indicate the high crystallinity of the sample. The average crystallite size of the sample calculated by applying Scherrer equation was 52 nm.

Figure 2.Indicates FTIR spectra of the prepared V_2O_5 sample. The V_2O_5 sample showed four main vibration modes in the 500-1010 cm^{-1} region. The terminal oxygen symmetric stretching vibration of V(V)=O was found at 1006 cm^{-1}(Phetmung, H., *et al*., 2008). The vibrations at 520 cm^{-1} and 815 cm^{-1} correspond to bridge oxygen symmetric and asymmetric stretching modes of V-O-V (Reddy, C.V.S., *et al*., 2008). The FTIR spectra also indicate that the compound contains only the elements V and O and vanadium exists as V(V).

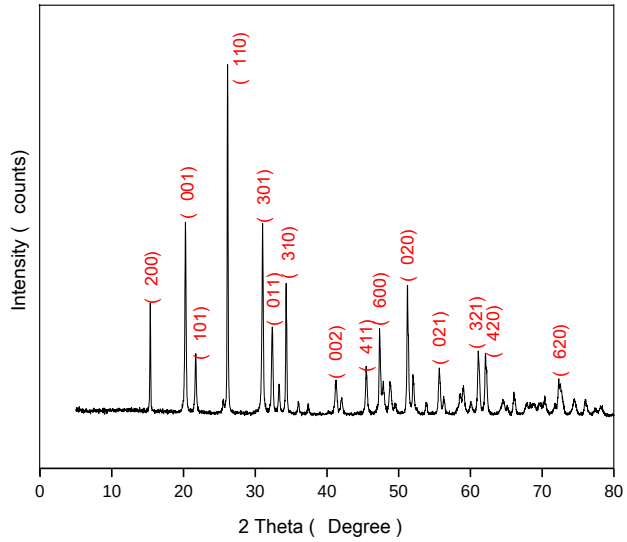

Fig. 1: XRD pattern of the prepared V_2O_5 sample

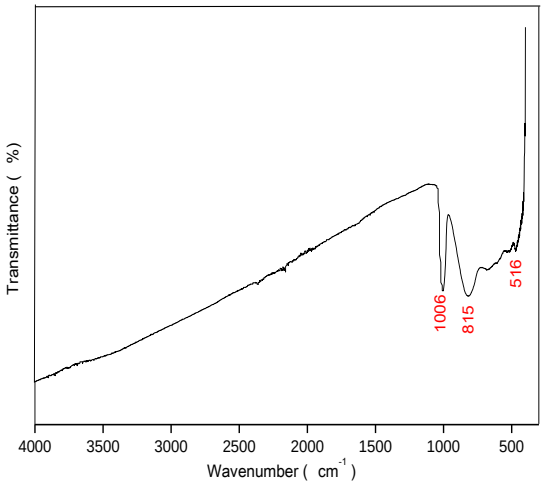

Fig. 2: FTIR spectra of the prepared V_2O_5 sample

Figure 3.shows the FESEM images of the prepared V_2O_5 sample at different magnifications. It is observed from the images that the sample consists of agglomerates with different shapes. The magnified images at 100 nm make it obvious that the structures are formed by the agglomeration of nano particles of V_2O_5. These particles are believed to exhibit in agglomerated morphology because of its ultrafine size (Chen, Y., *et al*., 2011). Large numbers of cavities are generated between the particles because of the release of gases such as CO_2 and NH_3 at the stage of thermal decomposition (Shahid, M. *et al*., 2010). The sphere like morphology of the V_2O_5 nanoparticles were clearly observed from the TEM images (fig.4). The sphere particles are highly dispersed with average size <20 nm.

The process of formation of vanadium pentoxide micro structures can be proposed as follows. When ammonium metavanadate was mixed with citric acid solution and dried, ammonium dimeric (citrato)dioxovanadium (V) $[(NH_4)_2[VO_2(C_6H_6O_7)_2]$ was formed. When it was thermally decomposed at 400°C, the complex decomposed to pure spherical V_2O_5 nanoparticles with the liberation of CO_2 and NH_3. As the reaction proceeds, the evolution of gases causes formation of voids or cavities between the particles and smaller particles aggregates to form bigger particles with aggregated morphology.

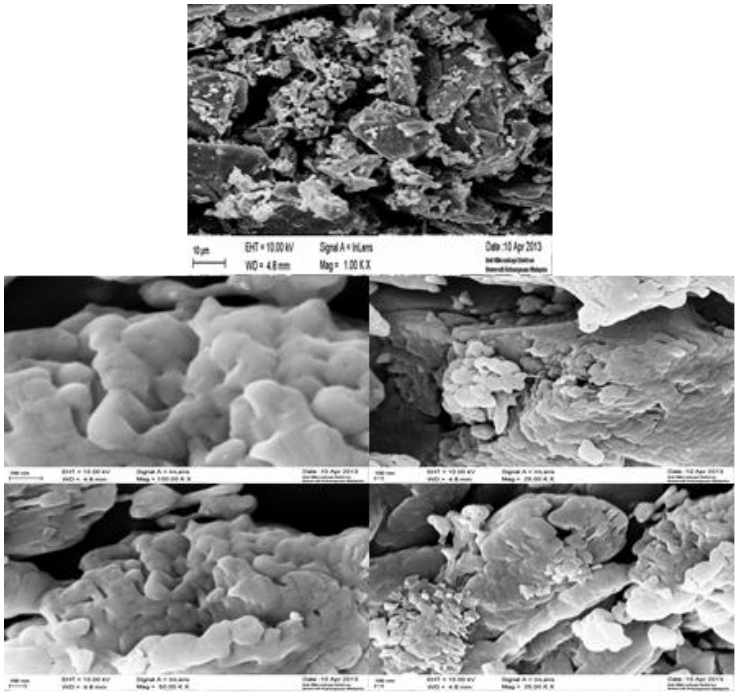

Fig. 3: FESEM images of the preparedV_2O_5 sample at different magnifications

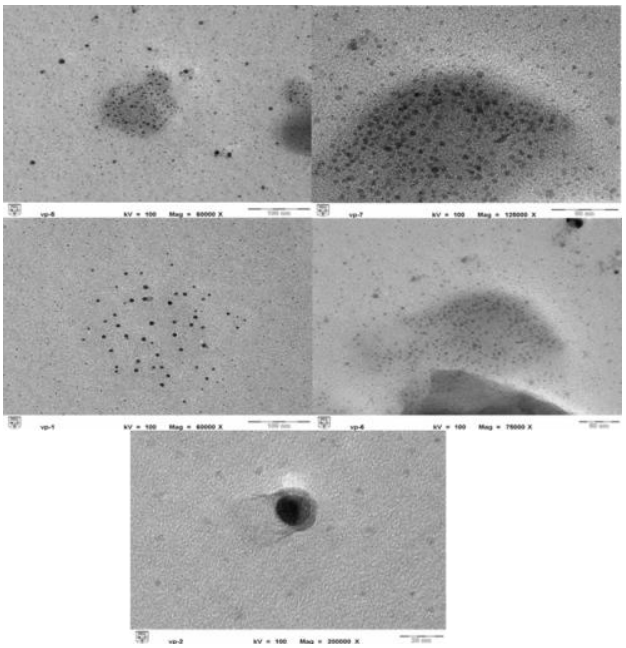

Fig. 4: TEM images of the prepared V_2O_5 sample at different magnifications.

Conclusion:

Single phased and highly crystalline micro V_2O_5 powder was prepared by simple solution mixing using citric acid as modifier. The prepared sample was phase pure as evident from XRD analysis. The agglomerated morphology of the prepared sample was observed from FESEM at lower magnification, but at higher magnification it became clear that the V_2O_5 powder consists of agglomerated V_2O_5 nanoparticles and the particles were found to be spherical in shape which was confirmed by TEM pictures. In conclusion we could provide a very simple, high yielding and low-cost method for the synthesis of highly pure micro V_2O_5 powder.

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