Structural and Morphological Charactrization of Nd Doped V_2o_5 Nanorods

D.Govindarajan, V.Uma Shankar

Abstract: ***V_2O_5 And Nd:V_2O_5 Nanorods Were Prepared By Using Low Cost Sol-Gel Method And The Synthesized Samples Were Analyzed Through XRD, SEM And HRTEM Techniques. The Structural Analysis Of The Processed V_2O_5 And Nd:V_2O_5 Samples Are Exhibited In The Form Of Orthorhombic V_2O_5 Phase. The SEM And HRTEM Images Of The Nd:V_2O_5 Sample Are Shown That The V_2O_5 Nonoparticles Appeared As Nonorods With Smooth Surface In Which Nd Spherical Particles Are Decorated.***

Keyword: ***V_2O_5 And Nd:V_2O_5 Nanorods, Sol-Gel Method, XRD, SEM And HRTEM Techniques.***

I. INTRODUCTION

Nanomaterials are elaborately studied because of their exclusive physical and chemical properties and its utilisation in distinct fields, which trigger to find the new synthetic methods for the preparation of nanomaterials. Recently, vanadium pentoxide (V_2O_5) has drawn more interest to the scientists for their remarkable properties and make them the vital element for a lot of modern technical and industrial applications. Since the scarcity of power generation, the electrode materials, maintaining the higher energy density, have been studied enormously. Vanadium pentaoxide is a normal intercalation composition since it possesses multivalent oxidation states (V^{2+}, V^{3+}, V^{4+} and V^{5+}) and the vivid structural chemistry, which allows redox-dependent characteristics [1 & 2].

Over the past few years, many of the scientists have been taken more effort to the synthesis of nanostructured V_2O_5 materials by the facial method, since the main characteristics of the V_2O_5 nanomaterials based on their composition, shape and hierarchical structures. Nanostructured V_2O_5 materials were prepared by different methods such as sol-gel, solvothermal, hydrothermal and microwave-irradiation methods [3]. Thus, these broad varieties of methods are preceded to produce various V_2O_5 nanostructures viz., nanotubes [4], nanobelts [5], nanorods [6], nanoneedles [7], nanosheets [8] etc. V_2O_5 nanomaterial shows the outstanding properties by controlling the parameters such as surface area, structure and morphology, which may be applied in vast industrial areas [9].

Previously, most of the researchers focused their study on rare earth ions doped V_2O_5 particularly V_2O_5 doped with Ce, Eu, Gd, Nd and Tb.

Nevertheless, an elaborate literature survey depicts only a few reports have been discussed the electrochemical analysis of rare earth ions (Ce^{3+}, Sm^{3+}, Eu^{3+}) intercalated with V_2O_5 nanostructure towards the supplant of the profitable and eco-toxic lead, mercury electrode materials for renewable storage batteries [10].

In this present work, V_2O_5 and Nd:V_2O_5 nanorods were prepared by using low cost sol-gel technique and the influence of Nd dopant on V_2O_5 crystal structure and microsturcture were evaluated through XRD, SEM and HRTEM techniques.

II. MATERIALS AND METHOD

A. MATERIALS

The entire chemicals used for synthesization are of analytical reagent grade and they are employed without any further refinement process. Ammonium metavanadate (NH_4VO_3), Neodymium oxide (Nd_2O_3) (Loba Chemical Pvt. Ltd.) and oxalic acid were purchased from Mark Pvt. Ltd.

B. SYNTHESIZATION OF V_2O_5 and Nd DOPED V_2O_5

V_2O_5 and Nd doped V_2O_5 nanoparticles were prepared by low cost sol-gel method. Initially, an appropriate quantity of oxalic acid was thoughly mixed in 50 ml of deionized water and heated at a temperature of 60 ºC. Then 7.018 g of NH_4VO_3 was gently mixed into the above solution. After 2 hours of stirring, the blue color gel was obtained and dehydrated at 80 ºC for 8 hours. Then the obtained powder was calcined at 400 ºC for about 4 hours, finally the yellow color V_2O_5 powder was obtained. A Similar procedure is ensued for the synthesisation of Nd doped V_2O_5 samples, by the addition of Nd_2O_3 (2%, 4%, 6% and 8%) with NH_4VO_3 - oxalic acid solution and designated as 2%Nd:V_2O_5, 4%Nd:V_2O_5, 6%Nd:V_2O_5 and 8%Nd:V_2O_5.

III.RESULTS AND DISCUSSION

A. XRD ANALYSIS

XRD spectra of the pure V_2O_5 and different percentages of Nd^{3+} ions (2, 4, 6 and 8%) doped V_2O_5 samples are shown in Fig. 1. From the XRD spectra, it observed that the peaks located at 2θ = 15.42º, 20.31º, 26.19º, 31.07º and 34.30º corresponding to the (200), (001), (110), (301) and (310) planes of V_2O_5 respectively. The diffraction peaks are matched to the orthorhombic structure of V_2O_5 (JCPDS 41-1426). Moreover, at lower Nd dopant percentages (2 and 4%), the orthorhombic phase of V_2O_5 is not affected and hence no secondary phases such as Nd or Nd_2O_3 was detected in the limit of the XRD technique. The orthorhombic phase and the non-appearance of contaminant peaks indicated that the prepared nanomaterials are well

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crystalline nature and also most of Nd^{3+} ions are merged into the V_2O_5 lattice. Also, the incorporation Nd^{3+} ions are evidenced from the position of the main peaks of V_2O_5 was slightly shifted to the lower 2θ values. This shift is provoked by the strain produced by the ionic radii of the Nd^{3+} and V^{5+} ions, respectively [11]. Further, at higher dopant percentage (6 and 8% of Nd), a small peak appeared at 2θ = 24.41° corresponding to neodymium oxide (Nd_2O_3) [12]. Since, the ionic radius of Nd^{3+} is (0.98 Å) 1.44 times higher than that of the ionic radius of V^{5+} ion (0.68 Å) and it was hard for Nd ion to replace the V^{5+} ion in the lattice of V_2O_5 due to the large disparity of their ion radius. This may be due to the diffusion of Nd element on the surface of V_2O_5 and appear in the form of Nd_2O_3 [11]. Collate to the XRD pattern of undoped V_2O_5, the peak intensities of Nd doped V_2O_5 decreases and the width of the diffraction planes becomes widen, which suggests that the crystallite size of V_2O_5 becomes lesser with Nd dopant percentage. Using Scherrer's formula (eqn.1), the mean crystallite size of the samples was calculated [13].

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

Here, D is the crystallite size, k is the Scherrer constant (0.89), λ is the wavelength (0.15406 nm) of the incident radiation, β is the FWHM and θ is known as a Bragg's angle. The average crystallite size was found to be 34 nm, 30.61 nm, 32.90 nm, 29.80 nm and 31.41 nm corresponds to the pure V_2O_5, 2%Nd:V_2O_5, 4%Nd:V_2O_5, 6%Nd:V_2O_5 and 8%Nd:V_2O_5 respectively. From the results, it is noted that the Nd ions prevent the growth of the crystallite size of the V_2O_5 nanorods.

B. FTIR ANALYSIS

Fig. 2 represents the FTIR spectra of V_2O_5 and 2%Nd:V_2O_5 nanomaterials. The four characteristic vibration modes appearing at 520, 642, 830 and 1016 cm^{-1} depicts the metal-oxygen bond of V_2O_5 matrix. From the Fig. 2, the band observed at 1016 cm^{-1} assigned to the V=O symmetric stretching mode is the evidence for the structural determination of orthorhombic V_2O_5 crystals. The band due to V-O-V symmetric stretching and asymmetric stretching modes was

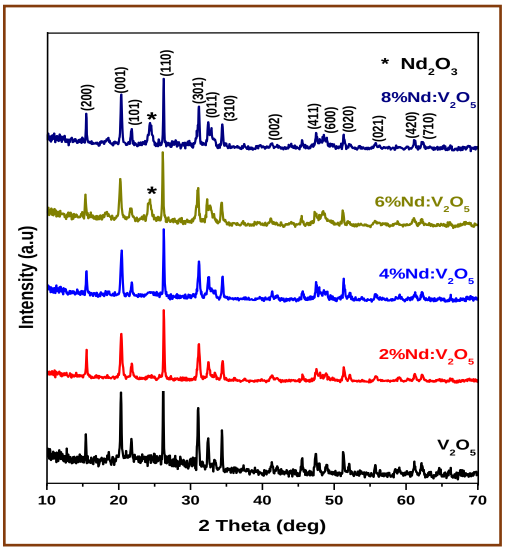

Fig. 1: XRD spectra of pure and Nd doped V_2O_5.

observed at 520 cm^{-1}, 642 cm^{-1} and at 830 cm^{-1}, respectively [13]. The peak around at 1016 cm^{-1} in the pristine V_2O_5 are shifted towards higher wavenumber in Nd doped V_2O_5 spectra, which indicates the appearance of dopant ions into the lattice, Nd^{3+} in the vicinage of oxygen and deformation of the V=O mode. The band at 642 cm^{-1} mode is moved towards lower wavenumber, which might be associated with the reduction of valence state from V^{5+} to V^{4+}.

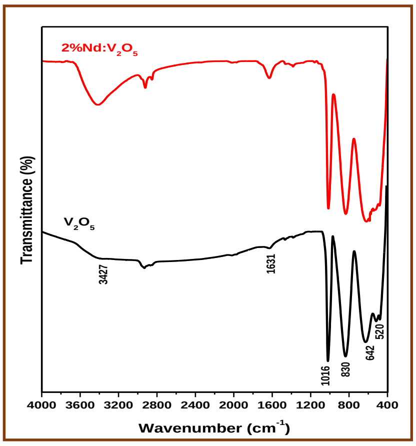

Fig. 2: FTIR spectra of V_2O_5 and 2%Nd:V_2O_5.

C. MORPHOLOGICAL ANALYSIS

Fig. 3 picturised the SEM images of pure and Nd doped V_2O_5 samples. The pristine V_2O_5 (Fig. 3a) has a distinct rod like shape with a smooth and uniform surface. The width of the nanorod is roughly 40-60 nm. The effect of Nd ions clearly reflects on the growth V_2O_5 nanorods are picturised in the Fig. 3b. Fig. 4 shows the HRTEM images of the samples. The pure V_2O_5 image (Fig. 4a) shows a large number of nanorods and the estimated width of the nanorod as ~ 35 nm. The HRTEM image (Fig. 4b) of the 2%Nd doped V_2O_5 sample was clearly noticed the Nd particles are attached with

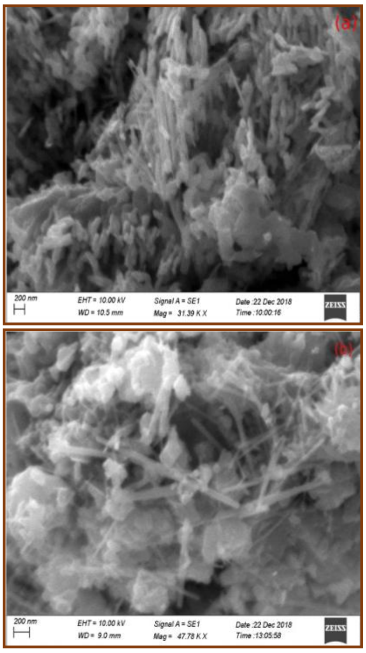

Fig. 3: SEM images of (a) V_2O_5 and (b) 2%Nd:V_2O_5.

V_2O_5 nanorods. As shown in Fig. 4c, the fringe patterns represent that the Nd doped V_2O_5 nanorods are in well crystalline nature. The observed interlayer distance around 0.37 nm corresponds to the (110) plane, which can be matched with XRD results.

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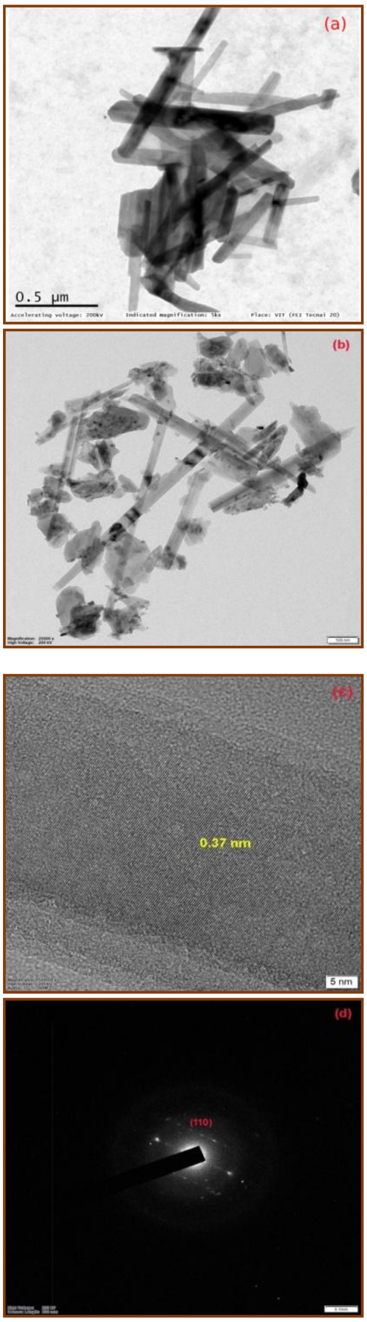

Fig. 4: HRTEM images of (a) V_2O_5 (b)2%Nd:V_2O_5 (c) high resolution image of 2%Nd:V_2O and (d) SAED pattern of 2%Nd:V_2O.

IV. CONCLUSIONS

In summary, pure and Nd doped V_2O_5 nanorod was effectively synthesized by simple sol-gel method. XRD study confirmed that the Nd:V_2O_5 nanorods have exhibited the pure phase of orthorhombic V_2O_5 and well matched with JCPDS card No. 41-1426. HRTEM and SEM studies confirmed that the Nd dopant is observed on the V_2O_5 nanorods.

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