Structural and Morphological Characterization of Nd Doped V₂O₅ Nanorods

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Abstract: V₂O₅ And Nd:V₂O₅ 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 V₂O₅ And Nd:V₂O₅ Samples Are Exhibited In The Form Of Orthorhombic V₂O₅ Phase. The SEM And HRTEM Images Of The Nd:V₂O₅ Sample Are Shown That The V₂O₅ Nanoparticles Appeared As Nanorods With Smooth Surface In Which Nd Spherical Particles Are Decorated.

Keywords: V₂O₅ And Nd:V₂O₅ 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 (V₂O₅) 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 pentoxide is a normal intercalation composition since it possesses multivalent oxidation states (V⁵⁺, V⁴⁺, V³⁺ and V²⁺) 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 V₂O₅ materials by the facial method, since the main characteristics of the V₂O₅ nanomaterials based on their composition, shape and hierarchical structures. Nanostructured V₂O₅ 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 V₂O₅ nanostructures viz., nanotubes [4], nanobelts [5], nanorods [6], nanoneedles [7], nanosheets [8] etc. V₂O₅ 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 V₂O₅ particularly V₂O₅ 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 (Ce³⁺, Sm³⁺, Eu³⁺) intercalated with V₂O₅ nanostructure towards the supplant of the profitable and eco-toxic lead, mercury electrode materials for renewable storage batteries [10].

In this present work, V₂O₅ and Nd:V₂O₅ nanorods were prepared by using low cost sol-gel method and the influence of Nd dopant on V₂O₅ crystal structure and microstructure were evaluated through XRD, SEM and HRTEM techniques.

II. MATERIALS AND METHOD

A. MATERIALS

The entire chemicals used for synthesis are of analytical reagent grade and they are employed without any further refinement process. Ammonium metavanadate (NH₄VO₃), Neodymium oxide (Nd₂O₃) (Loba Chemical Pvt. Ltd.) and oxalic acid were purchased from Mark Pvt. Ltd.

B. SYNTHESIZATION OF V₂O₅ AND Nd DOPED V₂O₅

V₂O₅ and Nd doped V₂O₅ 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 °C. Then 7.018 g of NH₄VO₃ was gently mixed into the above solution. After 2 hours of stirring, the blue color gel was obtained and dehydrated at 80 °C for 8 hours. Then the obtained powder was calcined at 400 °C for about 4 hours, finally the yellow color V₂O₅ powder was obtained. A Similar procedure is ensued for the synthesis of Nd doped V₂O₅ samples, by the addition of Nd₂O₃ (2%, 4%, 6% and 8%) with NH₄VO₃ - oxalic acid solution and designated as 2%Nd:V₂O₅, 4%Nd:V₂O₅, 6%Nd:V₂O₅ and 8%Nd:V₂O₅.

III. RESULTS AND DISCUSSION

A. XRD ANALYSIS

XRD spectra of the pure V₂O₅ and different percentages of Nd⁺³ ions (2, 4, 6 and 8%) doped V₂O₅ 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 V₂O₅ respectively. The diffraction peaks are matched to the orthorhombic structure of V₂O₅ (JCPDS 41-1426). Moreover, at lower Nd dopant percentages (2 and 4%), the orthorhombic phase of V₂O₅ is not affected and hence no secondary phases such as Nd or Nd₂O₃ 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...
crystalline nature and also most of Nd$^{3+}$ ions are merged into the V$_2$O$_5$ lattice. Also, the incorporation Nd$^{3+}$ ions are evidenced from the position of the main peaks of V$_2$O$_5$ 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$_2$O$_3$) [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$_2$O$_5$ due to the large disparity of their ion radius. This may be due to the diffusion of Nd element on the surface of V$_2$O$_5$ and appear in the form of Nd$_2$O$_3$, [11]. Collate to the XRD pattern of undoped V$_2$O$_5$, the peak intensities of Nd doped V$_2$O$_5$ decreases and the width of the diffraction planes becomes widen, which suggests that the crystallite size of V$_2$O$_5$ becomes lesser with Nd dopant percentage. Using Scherrer’s formula (eqn.1), the mean crystallite size of the samples was calculated [13].

$$D = \frac{k\lambda}{\beta \cos\theta}$$

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$_2$O$_5$, 2%Nd:V$_2$O$_5$, 4%Nd:V$_2$O$_5$, 6%Nd:V$_2$O$_5$ and 8%Nd:V$_2$O$_5$ respectively. From the results, it is noted that the Nd ions prevent the growth of the crystallite size of the V$_2$O$_5$ nanorods.

### B. FTIR ANALYSIS

Fig. 2 represents the FTIR spectra of V$_2$O$_5$ and 2%Nd:V$_2$O$_5$ nanomaterials. The four characteristic vibration modes appearing at 520, 642, 830 and 1016 cm$^{-1}$ depicts the metal-oxygen bond of V$_2$O$_5$ 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$_2$O$_5$ crystals. The band due to V-O-V symmetric stretching and asymmetric stretching modes was 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$_2$O$_5$ are shifted towards higher wavenumber in Nd doped V$_2$O$_5$ 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$^{4+}$.

### C. MORPHOLOGICAL ANALYSIS

Fig. 3 picturised the SEM images of pure and Nd doped V$_2$O$_5$ samples. The pristine V$_2$O$_5$ (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$_2$O$_5$ nanorods are picturised in the Fig. 3b. Fig. 4 shows the HRTEM images of the samples. The pure V$_2$O$_5$ 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$_2$O$_5$ sample was clearly noticed the Nd particles are attached with V$_2$O$_5$ nanorods. As shown in Fig. 4c, the fringe patterns represent that the Nd doped V$_2$O$_5$ 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.


REFERENCES


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IV. CONCLUSIONS

In summary, pure and Nd doped V2O5 nanorods was effectively synthesized by simple sol-gel method. XRD study confirmed that the Nd:V2O5 nanorods have exhibited the pure phase of orthorhombic V2O5 and well matched with JCPDS card No. 41-1426. HRTEM and SEM studies confirmed that the Nd dopant is observed on the V2O5 nanorods.