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Role of NaOH concentration on synthesis and characterization of β -V₂O₅ nanorods by Solvothermal method

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Abstract. In this work, synthesis of β -V₂O₅ nanorods by solvothermal method was adopted at optimum experimental conditions by varying the concentration of NaOH as 0.5M, 1M, 1.5M and 2M. The obtained products were characterized by following techniques, XRD, RAMAN, FTIR, and FESEM. The XRD results reveal the formation of β -V₂O₅ nanorods and its crystalline nature was confirmed by its lattice parameters. The Raman analysis shows a peak at 485, 770 and 998 cm⁻¹ corresponds to the vibration of O-V-O bonds. Similarly the FTIR peaks appeared at the 530 and 786 cm⁻¹ can be attributed to the symmetric stretching of Metal Oxide (V-O). The morphology and its structure of β -V₂O₅ nanorods can be analyzed by the FESEM techniques and its images shows the formation of β -V₂O₅ nanorods with different dimensions. The role of concentration of NaOH can be studied by comparing the FESEM micrographs. It is clearly evident from FESEM images as the concentration of NaOH increases from 0.5 M to 2M, the shape and size of the change rapidly and the surface effect results in the mesoporous features on the surface of nanorods. Its note worthy as the concentration of NaOH increases results in the flattening of nanorods could be observed due more of interaction of NaOH precursor solution.

INTRODUCTION

The Vanadium pentoxide (V₂O₅) nanoparticles have fascinated a great deal interest as an electrode material for energy storage devices and energy delivering sources. It exhibits properties of high output voltage and energy density for batteries and its handling is simple compare to the lithium ion batteries with cost effectiveness. It has been involved in good structural stability and layered structure includes benefit as the active materials for facilitating the more intercalation sites [1-5]. Cottineur et al. have investigated a comparative study on various nanostructured transition metal oxides to be used as an electrode for supercapacitors, in which V₂O₅ reported higher specific capacitance. Though there are few challenges to be addressed with V₂O₅ when used as an electrode material, like volumetric expansion, low strength, low conductivity and ionic diffusivity. Another problem is the Polarization influence the output voltage of the battery and results in the capacity fading [4]. The V₂O₅ as nanoparticles has significant in resolve the low conductivity and ionic diffusivity to certain extent [5]. Moreover the agglomeration and pulverization occurs in the formation of V₂O₅ nanoparticles has important role in its energy storage capacity. Metal oxides such as TiO₂, Fe₂O₃, and SnO₂ also have been extensively studied separately and its composition form with carbon materials as electrode materials [6–10]. Further there are so many literatures on transition bimetal oxides for different applications like battery and supercapacitor application were also be studied and evaluated [11-17]. Focuses on the simple and efficient metal oxide are dearly needed to address the current draw backs in the energy storage application. A composite of V₂O₅ / graphene has showed the improved performance in evaluation to individual units. Graphene/V₂O₅ composites are searched to great extent to capitalize their electrochemical properties as the electrode materials for supercapacitor applications [16-19].

MATERIALS AND METHODS

To synthesis β -V₂O₅ nanorods by varying the molar concentrations of reducing agent by adopting hydrothermal method was employed. The homogenous solution made up of 0.758 g Ammonium Meta vanadate nitrate NH₄VO₃ was dissolved in distilled water and stirred for 30 min. To this solution, 5 mM Sodium dodecyl sulfate (SDS) was added as shape directing agent, and the pH value of the precursor was measured and maintained at 2. The 0.5 M of NaOH was added drop by drop by above solution. The obtained homogenous precursor solution was transferred to 100 ml Teflon-coated autoclave and maintained at 180°C for 24 h. Then, the autoclave was allowed to cool down to room temperature naturally. The obtained powder was cleaned and washed by distilled water and ethanol three times to remove any untreated salt precursors and impurities. Then, the cleaned products were dried in hot air oven at 80°C overnight. The obtained samples were annealed at 600 °C and named SML1. The same procedure was followed for other two products by varying the concentration of NaOH by 1M, 1.5M and 2 M was named SML2, SML3 and SML4, respectively

X-ray powder diffraction (X'pert PRO analytical diffractometer) using CuK α as radiation (1.541 Å) source was carried out the phase and crystalline nature of prepared product. Imaging Spectrograph STR 500 nm focal length laser micro Raman spectrometer SEKI, Japan with resolution: 1/0.6 cm⁻¹/pixel and Flat Field: 27 mm (W) × 14 mm (H) was used. The morphology studies were carried out employing Scanning Electron Microscope (ZEISS-V80). Photoluminescence (PL) spectrum was measured using Varian Cary Eclipse Photoluminescence Spectrometer with Oxford low temperature LN₂ 77K setup. Infrared (IR) spectra were recorded using Fourier transform infrared spectrophotometer using Thermo Nicolet 380 with resolution 0.5 cm⁻¹ and S/N ratio: 2000:1 ppm for 1 minute scan.

RESULTS AND DISCUSSION

The XRD analysis was performed on the all synthesized products reveals the formation of well crystalline vanadium pent oxide (β -V₂O₅) nanorods. The peaks appeared at the 11.8°, 21.8°, 29.7°, 36.3°, 42.5° and 70.9° can be readily indexed to the (200), (110), (210), (101), (301) and (211) crystalline planes. The β -V₂O₅ nanorods exhibits the orthorhombic crystalline structure with lattice parameter a=11.54, b=4.383 and c=3.571 and the unit cell volume V= 1806.68 belongs to the Pmn21 space group. The crystalline size of obtained products SML1, SML2, SML3 and SML4 can be evaluated from the following equation. The Scherrer formula used to determine the crystalline size was given as $Dp = \frac{(0.94 \times \lambda)}{\beta \cos \theta}$, where Dp is the crystallite size, λ the wavelength of X-ray, β the wavelength of full width at half maximum, and θ the diffraction angle. The crystalline size of all products was 36.25, 28.56, 34.62 and 35.23 nm. The vibrational properties of β -V₂O₅ nanorods was analyzed by performing the Raman analysis shows the sharp peaks at the 485, 776 and 995 cm⁻¹. The peak at 485 corresponds to the bending vibration of V–O–V [20], and The stretching and bending modes of V–O generate peaks at 705 cm⁻¹ and the shift at 996 cm⁻¹ is due to stretching mode of V=O [21]. Since the Raman peaks appeared 485, 776 and 995 cm⁻¹ confirms the formation of the β -V₂O₅ nanorods. The FTIR studies executed for the synthesized β -V₂O₅ nanorods exhibits the sharp peaks at 530, 786, 1270 and 1630 cm⁻¹. The functional group presence in the synthesized nanomaterials was generally examined in the 4000-1000 cm⁻¹, where the peaks appeared below 1000 cm⁻¹ corresponds to the metal oxide vibrations modes in the obtained products. Therefore, the peaks at 530, 786 cm⁻¹ corresponds to the terminal oxygen symmetric stretching mode (vs.) of VLO and the bridge oxygen asymmetric and symmetric stretching modes [22]. Another peak at the 1270 and 1630 cm⁻¹ can be attributed to the presence of hydrated and non-hydrate vibration modes and the OH⁻ ions also can be noted from the peak at 1630 cm⁻¹. The Fig.4 (a-d) shows the FESEM images in 1µm scale for all synthesized products (SML1, SML2, SML3 and SML4). The micrograph clearly reveals the formation of β -V₂O₅ nanorods orientated in the different direction. Particularly at 0.5M of NaOH in the fig. 4(a) shows the complete nanorods originates from the single points configured into the flower like structure. The face of the nanorods consists of six face Hexa- structure and its ends have mesoporous arrangement. When the concentration of NaOH increase from 0.5m to 1M the nanorods flattening occurs due to the interaction kinetics of NaOH with the precursor solution. Similarly, the role of the SDS made impeccable function in the formation of nanorods. The conversion of nanorods to nanoplatelets occurs due to the increase in the concentration of NaOH. The fig 5(a-d) shows the images at 200 nm scale indicates the detail arrangement of β -V₂O₅ nanomaterials. It can be clearly observed from the FSESM images that, as the concentration of NaOH as reducing agent the configuration of β -V₂O₅ nanomaterials has varied tremendously that the nanorods originates from the particular point forms the flower like arrangements dislocated and form the nanostrips like structure orientated randomly in longitudinal axis. Similarly as the morality of the NaOH reaches to 1.5 M the nanostrips once again

deform and flattened into the nano rectangle shape can be evident from the FESEM micrographs. Nevertheless further increase in the concentration of NaOH as 2 M agglomerated all the nanostrips forms the irregular configuration of β -V₂O₅ nanomaterials which will immensely reduce the contact and surface area of synthesized nanomaterials in turns reflects in the aspect ratio of obtained β -V₂O₅ nanomaterials.

FIGURE 1. (a) XRD spectrum (b) Raman spectrum (c) Fourier transform infrared of β -V₂O₅ nanoparticles synthesized by a) 0.5 M NaOH, b) 1 M NaOH, c) 1.5 M NaOH and d) 2 M NaOH.

FIGURE 2. FESEM images of β -V₂O₅ nanoparticles synthesized by a) 0.5 M NaOH, b) 1 M NaOH, c) 1.5 M NaOH and d) 2 M NaOH

CONCLUSIONS

In this work, synthesis of β -V₂O₅ nanorods by solvothermal method was adopted at optimum experimental conditions by varying the concentration of NaOH as 0.5M, 1M, 1.5 M and 2 M. The XRD results reveal the formation of β -V₂O₅ nanorods and its crystalline nature was confirmed by its lattice parameters. The RAMAN analysis shows a peak at 485, 770 and 998 cm⁻¹ corresponds to the vibration of O-V-O bonds. The role of concentration of NaOH can be studied by comparing the FESEM micrographs. It is clearly evident from FESEM images as the concentration of NaOH increases from 0.5 M to 2 M, the shape and size of the change rapidly and the surface effect results in the mesoporous features on the surface of nanorods. Its note worthy as the concentration of NaOH increases results in the flattening of nanorods could be observed due to supplementary interaction of NaOH with the precursor solution and results in β -V₂O₅ nanomaterials which will immensely reduce the contact and surface area of synthesized nanomaterials in turns reflects in the aspect ratio of obtained β -V₂O₅ nanomaterials.

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