

Surfactant Free Synthesis and Study of Vanadium Pentoxide Nanostructure

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The varied oxidation state and layered structure are two importance features of vanadium pentoxide which makes it more special. Here, vanadium pentoxide nanostructure has been synthesized by a surfactant free and ecofriendly method using ammonium vanadate as a precursor salt. Synthesized nanostructure were characterized using X-ray diffraction method (XRD), Energy Dispersive X-Ray Analysis (EDAX), Scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and UV-Visible spectroscopy to study its structural, compositional, morphology, vibrational modes and optical behavior. XRD and FTIR confirm the orthorhombic phase of the vanadium pentoxide with a layered structure of irregular plates as observed from the SEM micrograph. Moreover, the band gap of material is 2.13 eV as evaluated from UV-Visible data.

Keywords: V₂O₅, DI water, Surfactant free.

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1. INTRODUCTION

Transition metal oxides due to their fundamental and technological aspect have become a material of greater interest. Vanadium pentoxide among them, possessing layered structure and various oxidation states with being thermodynamic most stable in vanadium-oxygen compound series is an excellent material [1]. It is a material with various important structural, chemical and optical properties and has a potential of being used in different application like in energy storage devices, gas sensors, as photo catalytic for waste water treatment, antimicrobial agent etc. [2-5]. Till now various physical and chemical approach methods have been developed with different vanadium salts and other chemicals for the synthesis of vanadium pentoxide.

Recently, Peijiang Cao and his group (2020) reported the synthesis of flower like V₂O₅ nanostructure by hydrothermal method using vanadium oxide, oxalic acid dehydrate, hydrogen peroxide and ethanol for the detection of xylene [6], while Reshma Radhakrishna and her group has synthesis nanostructure of V₂O₅ with morphology of irregularly arrange nanoplates by hydrothermal method using ammonium vanadate, nitric acid and distilled water for sensing of methane [7]. Karthik et al., in 2019, reported ultrasound assisted V₂O₅ nanoparticles using vanadium oxytrichloride, benzyl alcohol, ethanol and distilled water for the photocatalytic and antibacterial studies[8]. Similarly, different groups using methods like sol-gel method, chemical precipitation method, electrodeposition method, gel-combustion method, electrodepositing, chemical vapor deposition, etc. [9-14] has synthesis V₂O₅ nanostructure with different morphology for different application.

Here in this paper, we report a surfactant free, low

cost and ecofriendly way of synthesizing vanadium pentoxide nanostructure without using any complex system and as per our knowledge, it is first of its type in which vanadium pentoxide nanostructure is been synthesized using ammonium vanadate (AV) and distilled water as only chemical.

2. EXPERIMENTAL DETAILS

Ammonium vanadate (analytical reagent grade) was purchased from Merck-India. Precursor solution of 1 % W/V concentration was prepared by dissolving ammonium vanadate in triple distilled water. The prepared solution was stirred at 1000 rpm continuously for half hour at 60 °C so that AV decompose into vanadium oxide, ammonia and water



After that, the solution was heated at 100 °C till all the ammonia and water get evaporated with left over of mixed yellow-orange powder. This powder was annealed at 500 °C in furnace to get crystalline phase of V₂O₅.

3. RESULTS AND DISCUSSION

3.1 Structural Analysis

From the XRD pattern of synthesized nanostructure shown in Fig. 1, the diffraction peaks at 15.30°, 20.27°, 21.69°, 31.01°, 32.37°, 34.30°, 41.23°, 45.44° and 47.36° corresponding to (200), (001), (101), (110), (400), (011), (310), (002), (411) and (600) planes respectively are well matched with JCPDS Card no. 41-1426 and ref. [15]. No other phase peaks of vanadium oxide series was observed concluding in pure orthorhombic phase of V₂O₅.

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The lattice parameter as calculated from the three major peaks are $a = 11.52 \text{ \AA}$, $b = 3.57 \text{ \AA}$, $c = 4.37 \text{ \AA}$. The average crystallites size (D) of V_2O_5 nanostructure was calculated using Debye-Scherrer equation (1) and was found to be 50 nm.

$$D = \frac{0.9\lambda}{\beta \cos\theta}, \quad (1)$$

where λ indicates the wavelength of X-ray, 2θ is the position of peak in degrees, β is the FWHM in radians.

Moreover, the lattice strain (ϵ) and dislocation density (δ) [16, 17] were also calculated using equation (2) and (3) and were found to be $4.33 \cdot 10^{-3}$ and $4 \cdot 10^{14} \text{ lines/m}^2$

$$\epsilon = \frac{\beta \cos\theta}{4}, \quad (2)$$

$$\delta = \frac{1}{D^2}. \quad (3)$$

3.2 Compositional Characterization

To study the stoichiometry composition of the synthesized sample, energy dispersive analysis of the X-ray (EDAX) was used. The typical EDAX spectrum of synthesized V_2O_5 nanostructure is shown in Fig. 2. The spectrum consists of only vanadium and oxygen element peaks, confirming the purity of the synthesized V_2O_5 nanostructure.

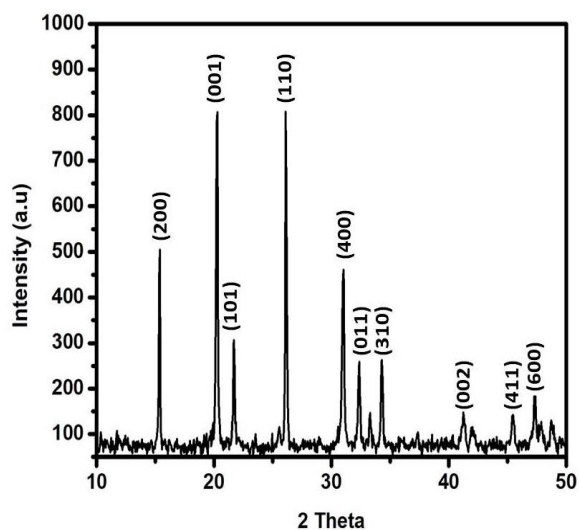


Fig. 1 – XRD pattern of V_2O_5 nanostructure

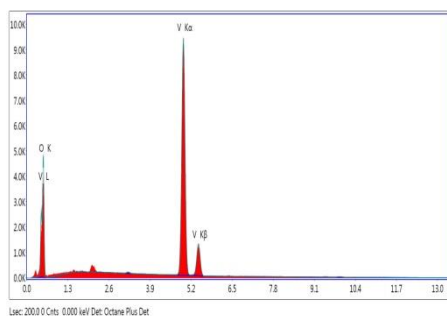


Fig. 2 – EDAX pattern of synthesized orthorhombic V_2O_5 sample

3.3 Morphological Analysis

Fig. 3 shows the SEM micrograph of the synthesized nanostructure. SEM images clearly reveals that the material is composed of irregular plate-like nanostructures with length in range less than $1 \mu\text{m}$ and thickness in the range of 100 nm to 200 nm. V_2O_5 nanostructure formed here are of smaller size as compared to the refs. [18, 19] having similar irregular arrangement of particles.

3.4 FTIR Analysis

The FTIR absorption spectrum ranging from 400 to 1400 cm^{-1} reveals that the absorption peaks at 493 cm^{-1} , 837 cm^{-1} and 1020 cm^{-1} are assigned to the characteristics of V–O vibration band. The peak at 493 cm^{-1} is attributed to V–O–V bridging bond [15] and the peak located at 837 cm^{-1} is assigned to the vibration of O–V₃ [20]. Whereas the orthorhombic structure of V_2O_5 resulted from the XRD data is confirmed by the absorption peak at 1020 cm^{-1} which is attributed to the symmetric stretching vibration of V⁺⁵=O band and it is the characteristic peak of the layered orthorhombic V_2O_5 structure [15].

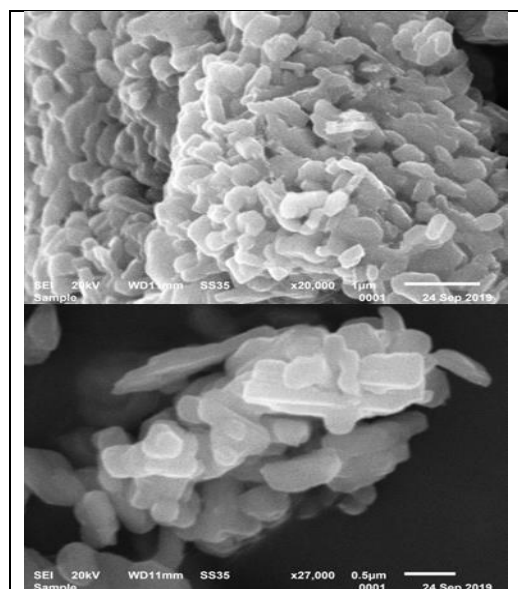


Fig. 3 – SEM micrograph of synthesized orthorhombic V_2O_5 sample

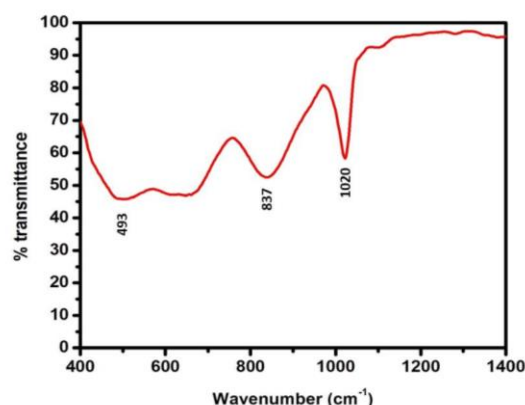


Fig. 4 – FTIR Profile of synthesized orthorhombic V_2O_5 sample

3.5 Optical Analysis

To study the optical property, diffuse reflectance of the sample was measured at room temperature over the wavelength range of 300 nm to 1200 nm as shown in Fig. 5. A sharp transmission edge is observed between 550 nm to 600 nm with the average reflectance around 55 %. With the help of Kubulka-Munk theory; we evaluated the optical band gap of the given sample using equation (4)

$$\frac{(1-R_{\infty})^2}{2R_{\infty}} = F(R) = \frac{k}{S}, \quad (4)$$

where $F(R)$ is the K-M function, k is the absorption coefficient, S is the scattering coefficient and R_{∞} is the reflectance of the sample at infinite thickness. The evaluated band gap is 2.13 eV, which is in good approximation with reference [21].

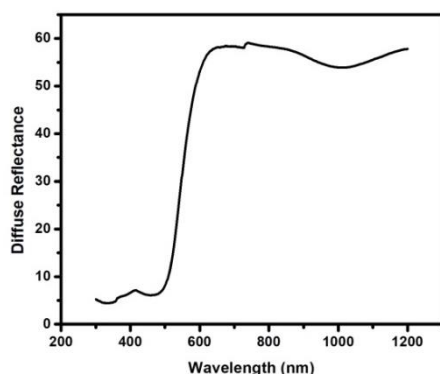


Fig. 5 – Diffuse reflectance spectra of synthesized orthorhombic V_2O_5 sample

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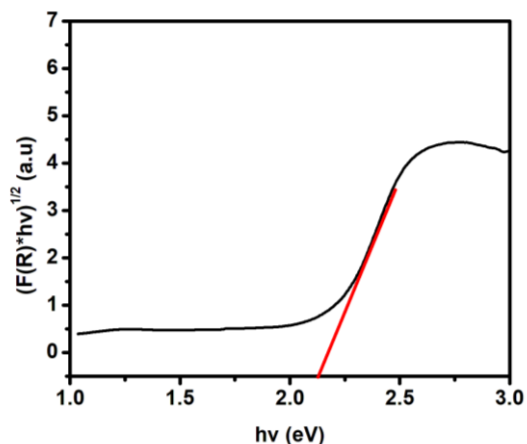


Fig. 6 – $(F(R)hv)^{1/2}$ versus hv shows band gap energy of synthesized orthorhombic V_2O_5 sample

4. CONCLUSIONS

We successfully report surfactant free, low cost and ecofriendly way of synthesizing V_2O_5 nanostructure without using any complex system. XRD and FTIR confirm the orthorhombic phase of V_2O_5 . SEM micrograph reveals the irregular arrangement of nanoplates. EDAX confirms the purity of the material and UV-Visible spectroscopy gives 2.13 eV band gap of the nanostructure.

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