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Synthesis and Characterization Studies of V_2o_5, Zro_2 Nanoparticles and V_2o_5-Zro_2 Nano composites

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***Abstract:*-- Metal oxides play a very important role in many areas of chemistry, physics and materials science in the Nano metric scale. In the present work, the metal oxides of Vanadium and Zirconium and their composites are synthesized and characterized. Vanadium pentoxide nanoparticles prepared by Precipitation method. Zirconium oxide nanoparticles prepared by Chemical method are used to synthesize V_2O_5-ZrO_2 Nano composites by Chemical method. All are characterized by UV-Vis studies, FTIR techniques; X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Atomic Force Microscope (AFM).The thermal behavior of the Nano composite is determined by Differential Scanning Calorimetry (DSC). From UV-visible spectrum of V_2O_5-ZrO_2nanocomposite the peak intensity isobestic point at 400nm which indicates that the intercalation of ZrO_2 in V_2O_5 lattices. XRD behavior of V_2O_5 nanoparticle, ZrO_2 nanoparticle and V_2O_5-ZrO_2nanocomposite exhibit the particle size distribution in the range of 7 to 85 nm. The structure of V_2O_5 nanoparticles, ZrO_2 nanoparticle and V_2O_5-ZrO_2nanocomposite are characterized using the SEM. Nano size of the V_2O_5 nanoparticle, ZrO_2 nanoparticle and V_2O_5-ZrO_2 nanocomposite are confirmed by AFM study.**

***Keywords:*-- Vanadium pentoxide, Zirconium oxide, Precipitation method, DSC, AFM**

I. INTRODUCTION

Accordingly the useful properties of vanadium oxides can be even more interesting in the nanometric scale. Nanostructured materials present unusual mechanical, electrical, optical and catalytic properties. They have been processed in thin film form and applied as optical and electrical devices. Vanadium oxide, as a wide band gap and n-type semiconductor material, is widely investigated because of its interesting electrochemical performance in lithium secondary batteries and its thermo chromic and electro chromic properties.

Zirconium oxide is a technologically important material due to its thermal and chemical stability and excellent mechanical properties, such as high strength and fracture toughness, high melting point, low thermal conductivity and high corrosion resistance. These unique mechanical and electronic properties of ZrO_2 ceramics have led to their widespread applications in the fields of structural materials, solid-state electrolytes, gas-sensing and corrosion-resistant.

II. EXPERIMENT DETAILS

A. *Preparation of V_2O_5 nanoparticles*

NH_4VO_3 was dissolved in hot distilled water. After a complete dissolution and removal of gaseous NH_3, concentrated nitric acid was added at 100°C. The reddish solution abruptly changed to brown precipitate. The resulting precipitate was collected by filtration, washed with water, and dried in a vacuum oven at 100°C for 24 hours.

$$2\ NH_4VO_3 \rightarrow V_2O_5 + 2\ NH_3 + H_2O$$

B. *Preparation of ZrO_2 nanoparticles*

ZrO_2 nanoparticles were prepared from reagent grade zirconyl chloride octahydrate ($ZrOCl_2.8H_2O$) and acetic acid (CH_3COOH). NH_4OH solution was used to keep the solution at pH = 5. Then, they were stirred for 2h at 40°C by using magnetic stirrer and aged for 24h at room temperature. The specimens were then removed from the deposition dispersion. After rinsing and drying, the ZrO_2 nanoparticles were obtained. Then the product was calcinated at 600°C for 2h.

C. *Preparation of V2O5 - ZrO2 nanocomposites*

V_2O_5 - ZrO_2 nanocomposite is prepared by Soft Chemical Method. In this method 100ml of 0.1M NH_4VO_3 is added to an aqueous solution of 200ml of 2M KOH and stirred well. To this mixture 100ml of 0.1M $ZrOCl_2.8H_2O$ is added, making the final volume of 400ml. The resulting mixture is stirred well and refluxed at an elevated temperature for 3 hours. The sample is filtered and washed with water to remove anions and dried at room temperature.

III. RESULTS & DISCUSSION

A. *UV-Visible spectroscopy*

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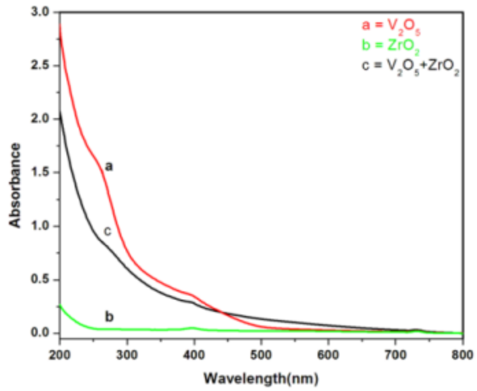

Figure 1: The UV-visible spectra of V_2O_5 (a), ZrO_2 (b) and V_2O_5-ZrO_2 mixture(c)

V_2O_5 has a shoulder at around 370-390 nm corresponds to the ligand to charge transfer and at 254 nm corresponds to ligand centered (LC) π-π* transition. The peak at 397 nm in ZrO_2 nanoparticle which corresponds to ligand charge (LC) transition. The absorption intensity of V_2O_5 decreases at 200 to 400 nm and compared with absorption intensity of V_2O_5-ZrO_2 mixture. Similarly the absorption of V_2O_5-ZrO_2 mixture increases from 435 to 700 nm with isobestic point at 440 nm. It indicates that V_2O_5 which intercalates with mixture. The absorption intensity at 397nm in V_2O_5-ZrO_2 mixture is decreased when compared with V_2O_5 and increased with ZrO_2 which indicates that there is ZrO_2 particles present in the V_2O_5 lattice sites.

B. FT-IR spectroscopy

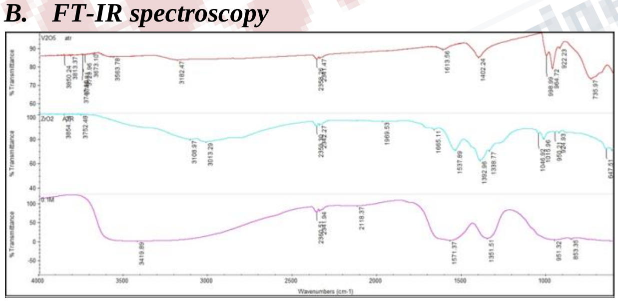

Figure 2: The stacked FT-IR spectrum of V2O5, ZrO2 and V2O5-ZrO2 mixture

The stacked FT-IR spectrum of V_2O_5, ZrO_2 and V_2O_5-ZrO_2 mixture is shown. The peak at 964 cm^{-1} for V_2O_5 [1,2] is due to V-O-V stretching and which is shifted to lower frequency with broad band at 951cm^{-1} in V_2O_5-ZrO_2 composite which indicates that there is a Zr-O-V stretching. The peak at 1402 cm^{-1} for V_2O_5 and peaks at 1339 cm^{-1} and 1392 cm^{-1} for ZrO_2 are merged together and shifted to lower frequency with broad band at 1351 cm^{-1} for V_2O_5- ZrO_2 composite. This indicates both the metal oxides are mixed to form nanocomposite.

C. XRD study

The particle size was calculated from XRD data using Scherrer's formula D= 0.94 x λ / β cos θ

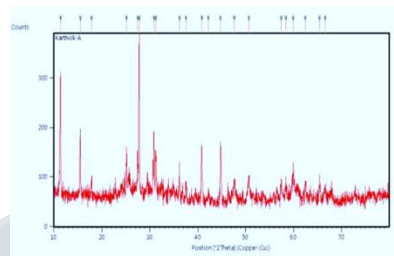

Figure 3a: XRD spectrum of V2O5 nanoparticle

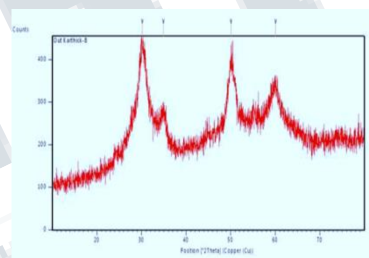

Figure 3b: XRD spectrum of ZrO2 nanoparticle

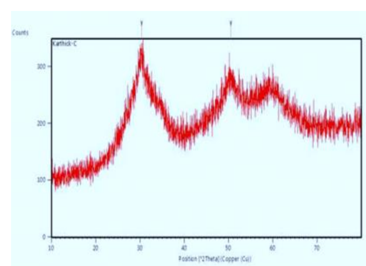

Figure 3c: XRD spectrum of Nano composite

The X-ray diffraction pattern for V_2O_5 nanoparticle shows that the high intensity peaks at the range 2θ value from 11.3° to 44.8°. The particle size is calculated as 85 nm using Scherrer's formula.The X-ray diffraction pattern for ZrO_2 nanoparticle shows that the high intensity peaks at the range 2θ value from 30.1° to 60.1°. The particle size is calculated as 55 nm.The X-ray diffraction pattern for V_2O_5-ZrO_2 nanocomposite shows that the high intensity peaks at the range 2θ value from 30.3° to 50.5°. The particle size is 7nm.

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D. Scanning Electron Micrograph (SEM) study

The structure of V_2O_5 is **flower** like in nature.[3] ZrO_2 nanoparticles exhibit moderately **fibre** like structure. V_2O_5-ZrO_2nanocomposite exhibits moderately **fibre like** structure.

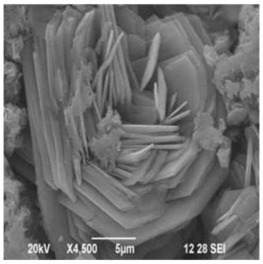

Figure 4a: SEM Images of V2O5 nanoparticle

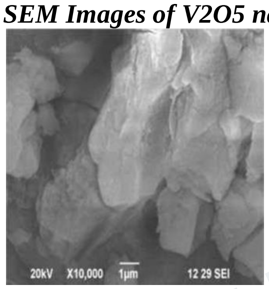

Figure 4b: SEM Images of ZrO2 nanoparticle

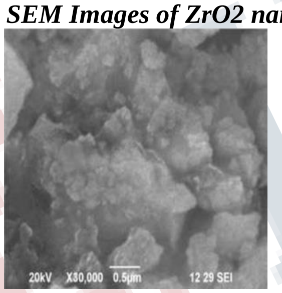

Figure 4c: SEM Images of nanocomposite

E. Differential Scanning Calorimetry (DSC) study

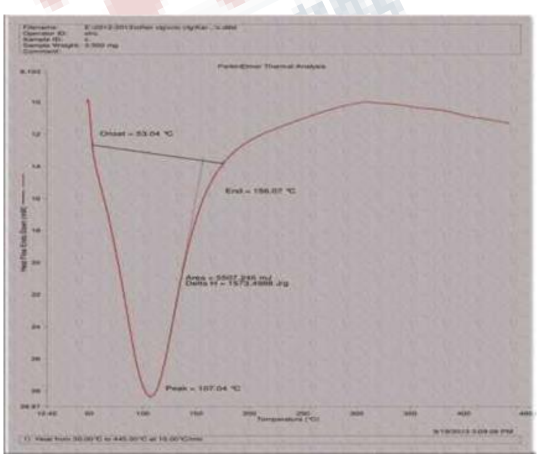

Figure 5: DSC thermogram of the nanocomposite

- Glass transition temperature (Tg) = 58^0C
- Crystallization transition temperature (Tc) = 55^0C
- Melting point (Tm) = 107.04^0C

F. Atomic Force Microscope (AFM) study

The AFM images of V_2O_5, ZrO_2 nanoparticle and V_2O_5-ZrO_2 nanocomposite are shown below. The V_2O_5 nanoparticle size is in the range of 55 to 200 nm. The ZrO_2 nanoparticle size is in the range of 60 to 100 nm and the V_2O_5-ZrO_2 nanocomposite size is in the range of 20 to 50 nm. From the size of the particles, we concluded that V_2O_5 nanoparticles**,** ZrO_2 nanoparticle, and V_2O_5-ZrO_2 nanocomposite are in the nanoparticle range.

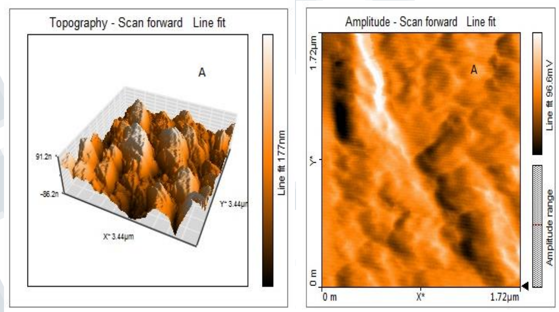

Figure 6a: AFM images of V2O5 nanoparticle

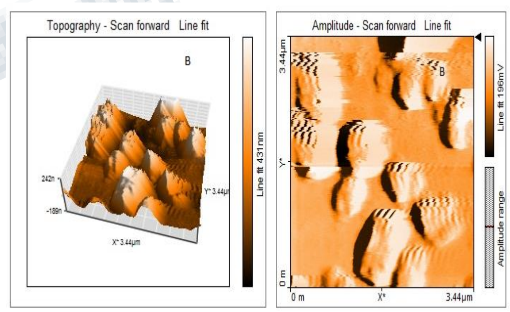

Figure 6b: AFM image of ZrO2 nanoparticles

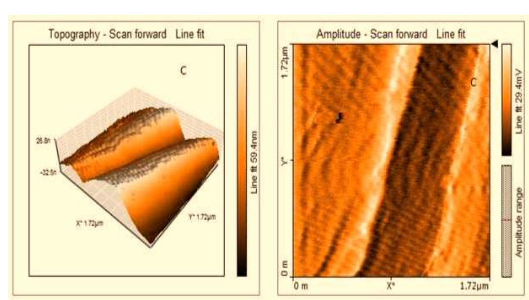

Figure 6c: AFM images of V2O5-ZrO2 nanocomposite

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IV. CONCLUSION

V_2O_5 nanoparticle, ZrO_2 nanoparticle, V_2O_5-ZrO_2nanocomposite are synthesized by precipitation and chemical methods and characterized using various spectral techniques. From the UV-visible spectrum of V_2O_5-ZrO_2nanocomposite the peak intensity decreases from 200-400 and increases from 400-800 nm with isobestic point at 400nm which indicates that the intercalation of ZrO_2 in V_2O_5 lattices .The FTIR spectral results revealed that the V_2O_5-ZrO_2 nanocomposite the V-O, Zr-O band is shifted to lower frequency. XRD behavior of V_2O_5 nanoparticle, ZrO_2 nanoparticle and V_2O_5-ZrO_2nanocomposite exhibit the particle size distribution in the range of 7 to 85 nm. From the SEM analysis V_2O_5 nanoparticles exhibits flower like structure, ZrO_2 nanoparticle exhibits fibre like structure and V_2O_5-ZrO_2nanocomposite exhibits smooth fibre like structure. DSC studies revealed the thermal stability of V_2O_5-ZrO_2nanocomposite. The values of Tg, Tm and Tc are calculated from DSC thermogram and indicates that there is a uniform mixture of V_2O_5 and ZrO_2. AFM study confirms the nano size of the V_2O_5 nanoparticle, ZrO_2 nanoparticle and V_2O_5-ZrO_2 nanocomposite.

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