Vanadium Pentoxide Thin Films: Synthesized by Thermal Decomposition, Characterization and Application as a Gas Sensor

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ABSTRACT

Vanadium pent-oxide nanoparticles were synthesized by thermal decomposition technique for sensing harmful gases in the atmosphere. In this synthesis citric acid was used for reducing the size of the vanadium pentoxide particles. The vanadium pentoxide nanorods are formed in this synthesis. vanadium pentoxide nanoparticles were coated on glass substrate by spin coating techniques. Various characterization techniques likes XRD, SEM, TEM, FTIR, TGA have been done and GAS SENSOR ANALYSIS has to been done. XRD results have shown the size of the crystalline and morphology. Vanadium pentoxide thin films were used to sense CO, NH3, Methane and acetone.

Keywords: thermal decomposition, spin coating, vanadium pentoxide

1. INTRODUCTION

Gas sensors plays a vital role for maintaining clean and green environment. This has got its application in food processing industry, chemical industry and the industries where various affluents are emitted. These sensors when reduced to nano scale show increase in their conductivity property due to significant increase in their surface area to volume ratio. These sensors help us in measuring, monitoring and controlling the concentration of the toxic gases in our surroundings. These also consume very less amount of power, simple and are portable.

These are the papers on vanadium oxide nanoparticles till date. Vanadium oxide have been studied since the discovery of VO nanotubes by Nesper and co-workers (Bachmann et al. 1961; Gopinath and Patel 2000; Raible et al. 2005; Spahr et al.1998; Nesper and Muhr 1998). Outstanding structural, optical and electrical properties which were calculated in "study of structural, electrical and optical properties of vanadium oxide condensed films deposited by spray pyrolysis technique"(M. Mousavi A. Kompany N. Shahtahmasebi, M. M. Bagheri-Mohagheghi in 2013), the vanadium

oxides have found many potential applications in lithium ion batteries which were considered in "Facile synthesized nanorod structured vanadium pentoxide for high-rate lithium batteries"(Anqiang Pan, ab Ji-Guang Zhang, Zimin Nie, Guozhong Cao, Bruce W. Arey, Guosheng Li, Shu-quan Liang and Jun Liu in 2010). For example, V2O5 nanofibres show higher sensitivity than other oxides (e.g., SnO2, TiO2, and MoO3) to organic amines (e.g., 1-butylamine with limit of detection below 30 ppb) that are important for analysis in food industry and medical diagnosis (Raible et al. 2005; Eranna et al. 2004).

2. MATERIALS AND SYNTHESIS METHOD

Materials: Vanadium pentoxide (V_2O_5) AR grade(99.5%), Citric acid($C_6H_8O_7$), Poly vinyl pyrrolidone(PVP), hydrogen peroxide(H_2O_2)

Method: The synthesis of vanadium (V) oxide nanoparticles was done by thermal decomposition method. 30ml of de-ionized water was taken and 3.83gm of citric acid and dissolved till a homogenous solution was formed. After this 0.6gm of vanadium pentoxide was added to the above solution and then it is stirred at 500 rpm at 30^{o}C till the solution changes from orange to yellow to blue color. Further this solution is heated at 80^{o}C for 2 hr. This heating process was continued till the water gets evaporated and is crushed to powder. Next this powder is calcinated for 2 hr at 450^{o}C. The change of color of the powder from blue to dark orange is the indication of formation of the vanadium pentoxide nanoparticles.

Thin films formation:

The vanadium pentoxide nanoparticles are dissolved in hydrogen peroxide. Dissolution of V2O5 in H2O2 was highly exothermic. So 0.5gm of V_2O_5 was dissolved in 30ml of 15% H_2O_2 solution. Then 2.5gm of PVP is added. Subsequently this solution was stirred for 4hr at 80^{o}C till a gel solution is formed. After this the solution is coated on a glass substrate by spin coating.

3. CHARACTERIZATION TECHNIQUES

Gas sensor which was prepared from vanadium pentoxide nanoparticles has undergone various characterizations like XRD, SEM, TEM, FTIR and TGA.

XRD:- The structure of crystal, phase of the sample, average crystallite size, lattice parameters and strain determined by X-ray diffraction using Bruker D8 advanced X-ray diffractometer using

Cu$K\alpha$ radiation.

SEM:- The shape and the microstructure is confirmed by FEI Quanta 400 with EDS with an accelerated voltage of 25KV.

TEM:- TEM was used inspecting crystalline structure by using FEI Tecnai G2.

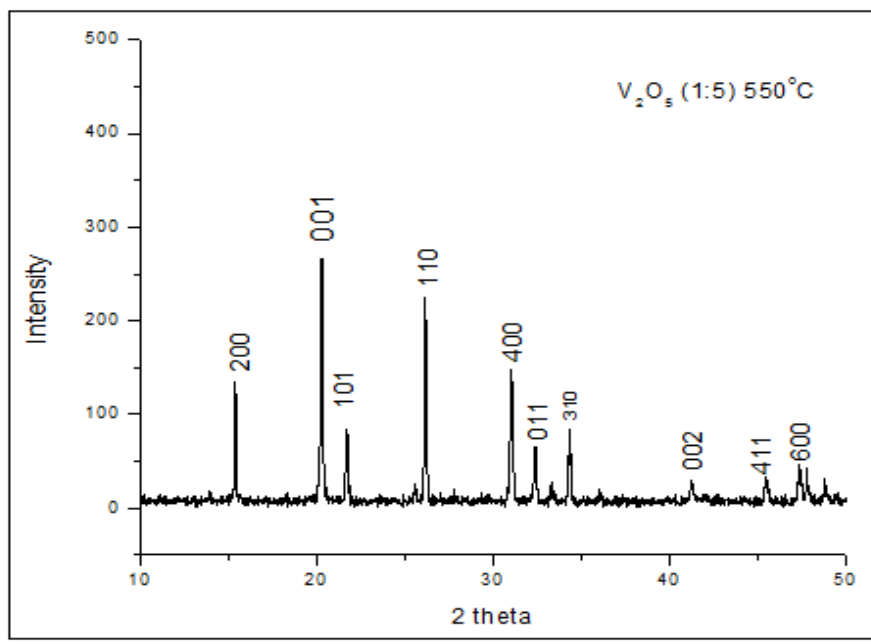
FTIR:- The bond analysis/participation of elements in the reaction by BRUKER OPTICS FTIR Instrument.

TGA:- . The complementary information obtained allows differentiation between endothermic and exothermic events with no TG/DTA-6300 thermal analyzer.

4. RESULTS AND DISCUSSIONS

XRD:-

The powders were characterized by X-ray diffraction (XRD) using a Bruker D8 advanced X-ray diffractometer using Cu*Kα* radiation. All the samples are scanned in a range of 10 to 50°(2 theta) with a step size of 0.02. The obtained sample has no other peaks indicating high purity of the sample. According to the Scherrer method the crystallite size is found to be 56.21nm and that of the commercial V2O5 is 119.54nm. The extended peaks are representing the dimensions of the nano range particles. Peaks are observed at 15, 20, 22, 27, 32, 34, 41, 45, 48, 51 respectively corresponding to the (h k l) values of the peaks (200), (001), (101), (110), (400), (011), (310), (002), (411) and (600) respectively



SEM:-

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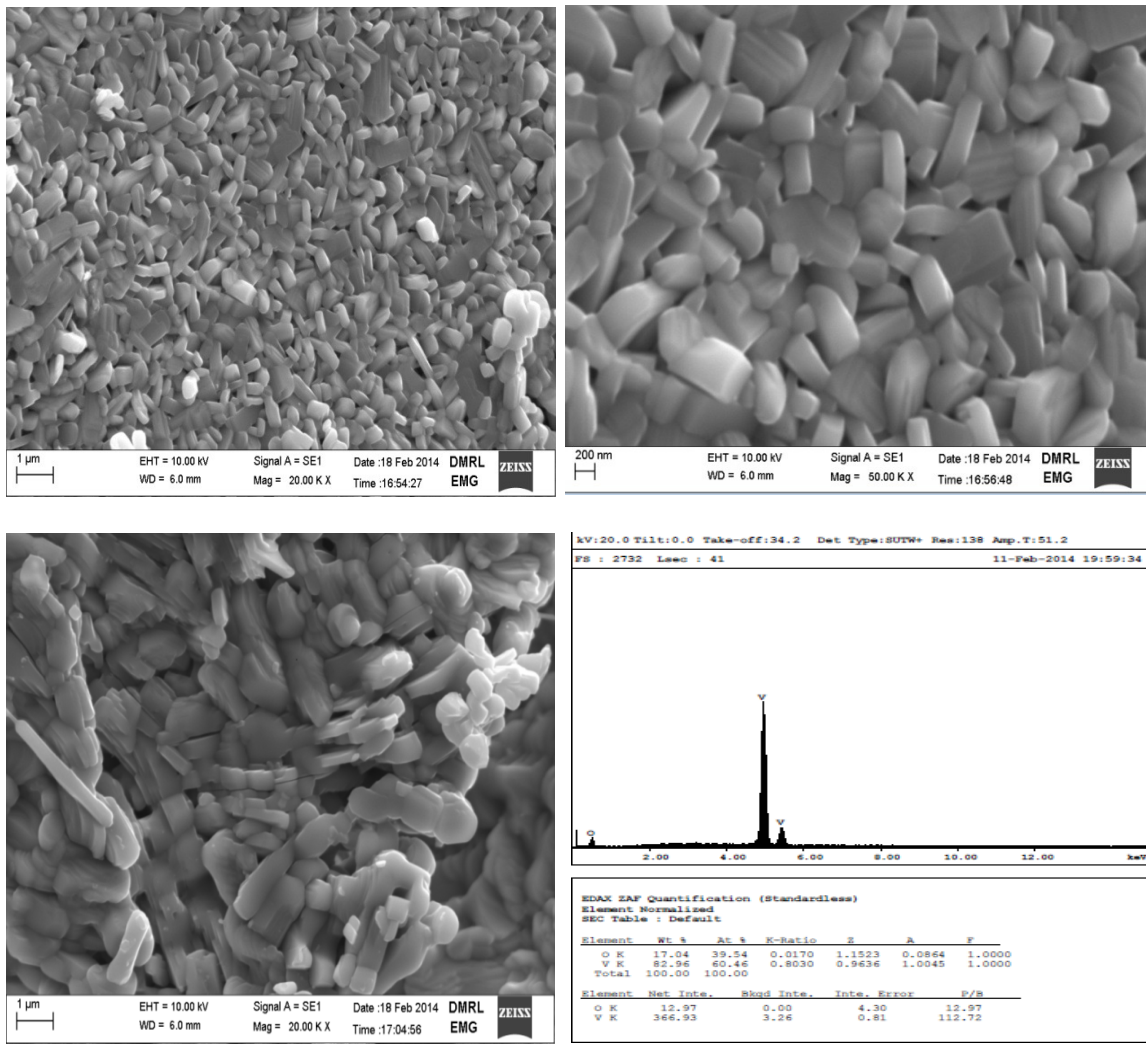


iii a b

Fig 2:a)SEM images of the vanadium pentoxide particles and b) EDS of the vanadium pentoxide

From the above figure 2 a) i) orthorhombic structure of the compound is determined and there is uniform particle size distribution. From figure 2 a) ii) This shows the clear crystalline nature of the nanostructures this is obtained at 50.00K magnification iii) agglomerization of the layers are shown at 20.00K magnification.

From figure 2 b) the weight percentage of vanadium present in the sample is 82.96% and that of oxygen is 17.04%. Atomic percent of vanadium in the sample is 60.44% and oxygen is 39.66%.

TEM:-

From the figure a) orthorhombic structure of the sample is formed. In fig b) spheres are formed due to the agglomerization of the layers.

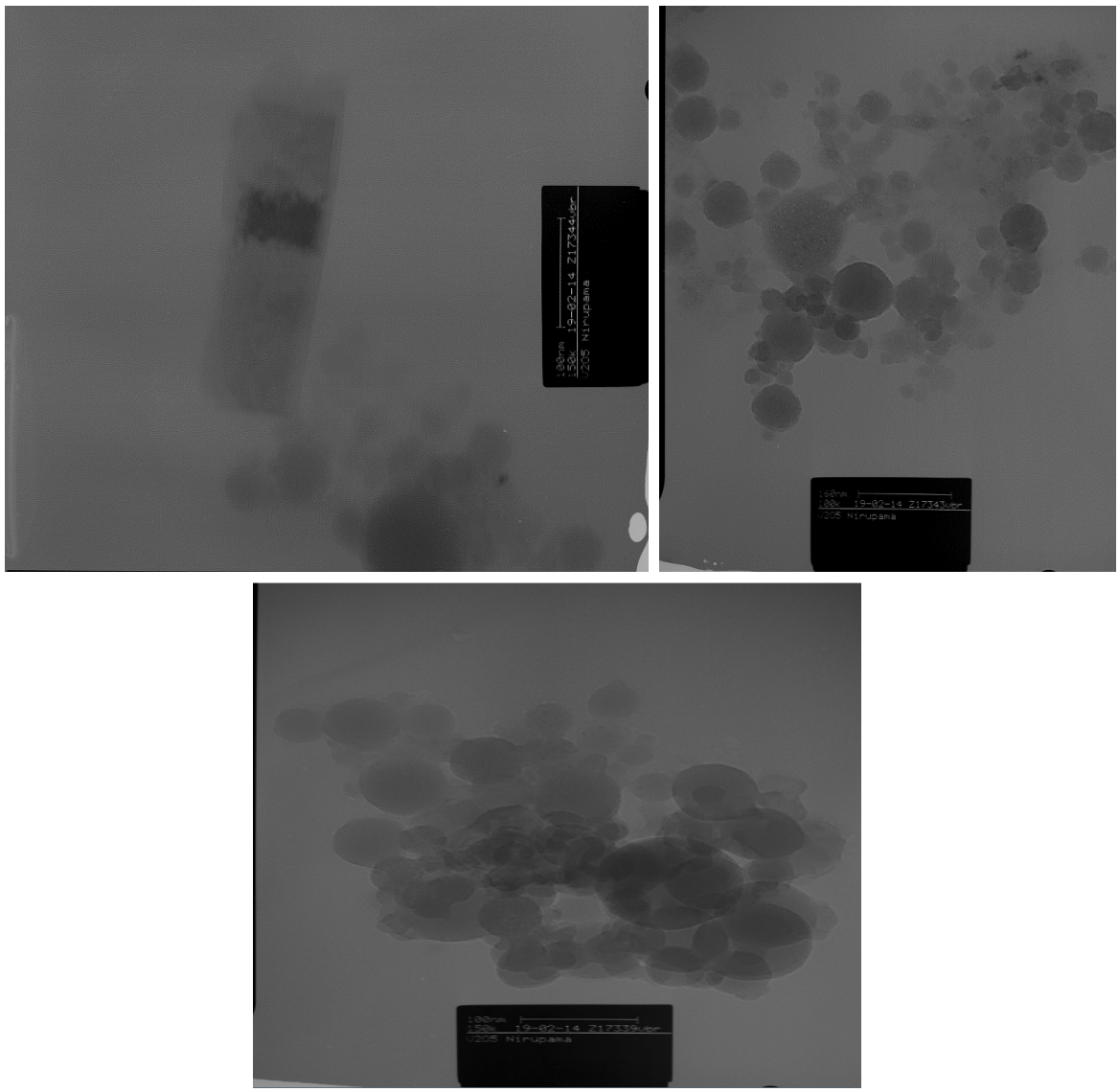


Fig 3

FTIR:-

The FTIR spectrum of vanadium oxide nanotubes exhibits the N-H vibration of NH^{+3} at 3431cm^{-1} and 3412cm^{-1}. Various signals between 1050cm^{-1} to 500cm^{-1} are attributed to different V-O type bonds. Vanadium pentoxide nanotubes are showing very intense peaks at 1020cm^{-1} and 837cm^{-1}.

From the fig. it is seen that for vanadium and oxygen bonds three kinds of absorption spectrum are seen at 1020cm^{-1}, 837cm^{-1} and 617 cm^{-1}. The spectrum at 1020 cm^{-1} is due to the presence of V=O, vanadyl group. The spectrum at 835cm^{-1} shows absorbance due to the presence of O -(V)3. The value at 617cm^{-1} shows the absorbance due to V -O -V group.

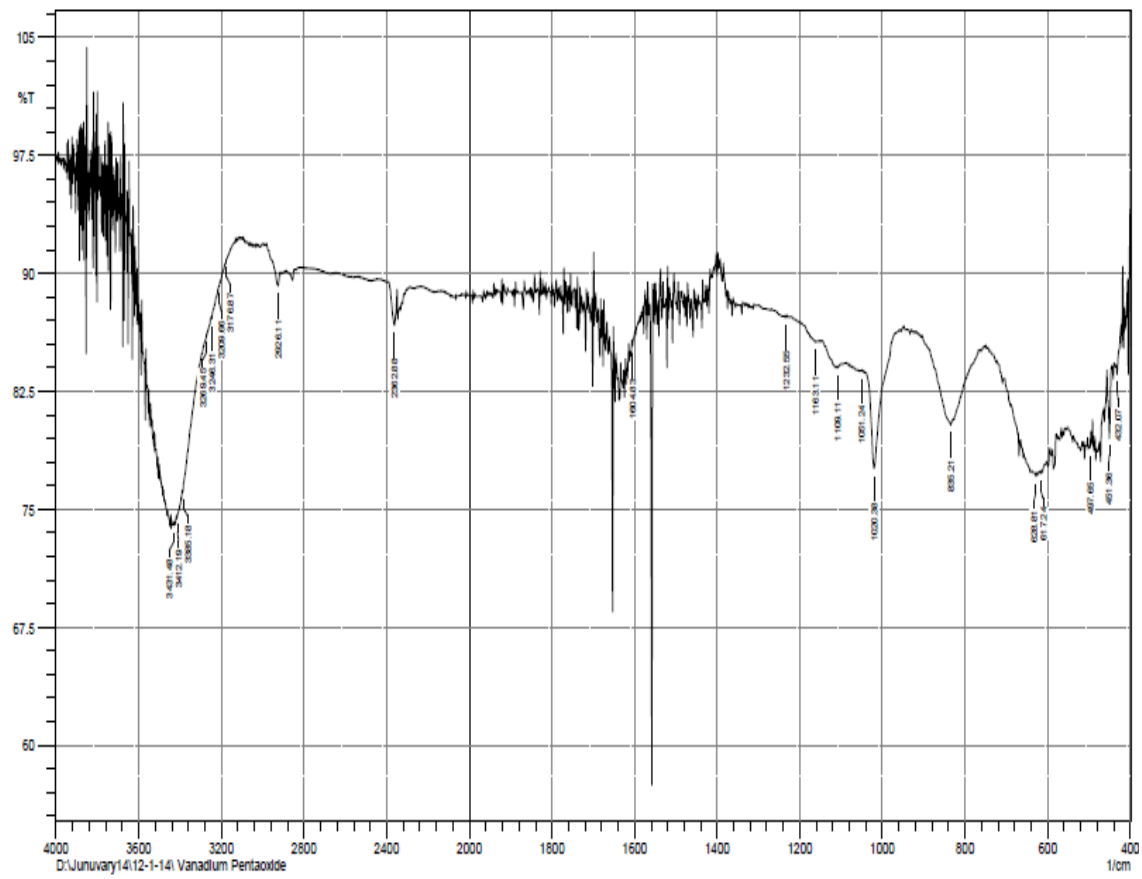
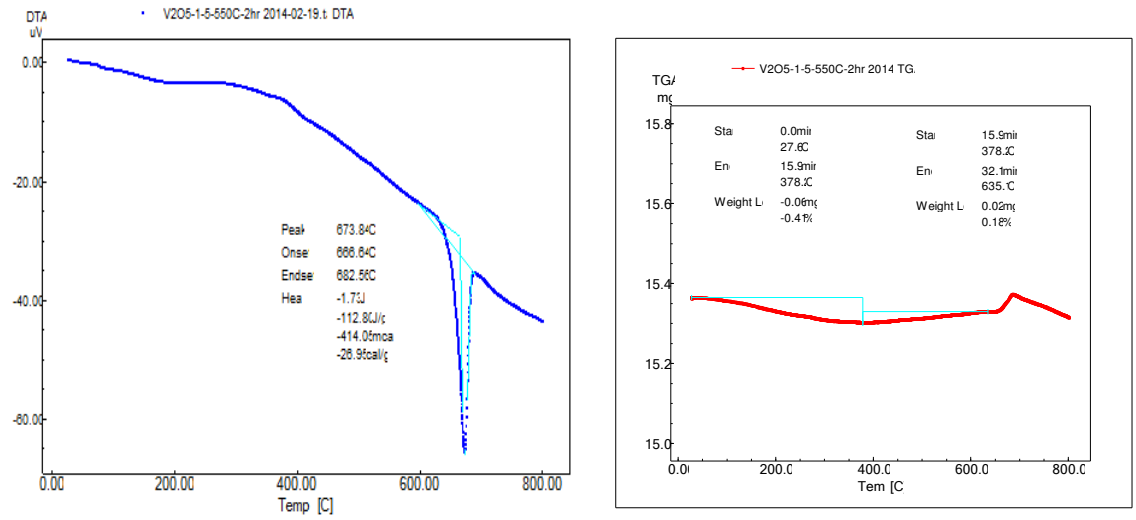


Fig 4:FTIR spectrum of vanadium pentoxide nanostructures.

TGA:-



a b

Fig 5: DTA of vanadium pentoxide nanostructures and TGA of vanadium pentoxide nanostructures.

From the above TGA result, vanadium pentoxide presence is confirmed. A small weight loss is observed at 200^{o}C which indicates the presence of the vanadium pentoxide and also a small weight loss is observed at 600^{o}C due to the formation of V^{+4} ions from V_2O_5.

5. CONCLUSION

Effectively, vanadium(v) oxide nanoparticles were synthesized by a thermal decomposition. The obtained product was confirmed to be vanadium(v) oxide, the crystal structure was studied and the crystallite size of 56nm of V_2O_5 nanoparticles was studied using XRD.The functional group in the V_2O_5 were studied using FTIR spectroscopy, V=O bond was observed at 1020cm^{-1}, O-(V)^3 bond at 835 cm^{-1} The weight loss of 0.182% was observed by the sample, which is found out by TGA and the heat flow is found by using DTA. SEM and TEM showed that nanostructures were formed and there was agglomerization of layers. Orthorhombic structure was seen in these characterizations.

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