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Low Temperature Synthesis and Characterisation of Nanosized Vanadium Oxide

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ABSTRACT

Self propagating low temperature synthesis method for the synthesis of oxide nano materials integrates synthetic chemistry. The combustion process need an efficient fuel for complete conversion of the reaction mixture in to crystalline product Present work reports, synthesis of vanadium oxide by vanadium oxalate precursor employing polyvinyl alcohol as a fuel. The structure of as synthesised oxide is characterised by X-ray diffraction (XRD), bonding by Fourier transfer infrared (FTIR), morphology and particle size by Scanning Electron Microscope (SEM) tools. Crystallite sizes and density measurement of the sample is under taken. The crystallite sizes were calculated using X-ray line broadening and density measurements were under taken by various methods. Crystalline behaviour is observed by XRD pattern and vanadium – oxygen (V-O) bond formation was confirmed by FTIR study.

Keywords: Self-propagating, Structure, Bonding, Morphology, Crystallite size, Density.

INTRODUCTION

Oxide materials have created a lot of interest in recent years due to its specific properties and potential applications. Ceramic material of the present generation is so important because of its properties [1,2]. Due to technological importance, many researchers attracted lot for the synthesis of oxide materials at nano dimension [3,4]. Interest has increased following the observation that the properties are strongly dependent on the size of particles with dramatic changes when nanometric sizes are acquired [5]. The new synthetic routes for preparation of nano structured materials are under constant investigation and low temperature synthesis method seems to be simple [6]. Among the various oxides, Vanadium oxide is an important materials finds various applications in different fields. Hence, much attention is given for the preparation and characterisation of the vanadium oxide material. The synthesis of vanadium oxide nanoparticle using vanadium carboxylate precursor employing combustion synthesis method is reported in the present study. Polyvinyl alcohol (PVA) is used as a fuel for the conversion of vanadium oxalate in to its oxide nanoparticles. Polyvinyl alcohol is a good surfactant and dispersant, hence the precursor will be well dispersed in the molten PVA which is needed for the initiation of combustion process. Initially it burns partially with fuel and it completes conversion of precursor in to cadmium oxide particles at high temperature. The prepared sample is well characterised for its structure by X-ray diffraction (XRD),

morphology by Scanning Electron Microscope (TEM) and bonding by Fourier Transform Infrared study (FT-IR) techniques. Crystallite size and various densities of vanadium oxide are calculated.

MATERIALS AND METHODS

Vanadium salt, oxalic acid and ammonia chemicals are used in the present study were of AR grade. Polyvinyl alcohol of molecular weight 125,000 was obtained commercially is used as fuel. Thermal decomposition method is adopted for the synthesis of vanadium oxide nanoparticles

Preparation of cadmium oxalate precursor : The hydrated vanadium oxalate precursor was prepared by dissolving equimolar proportions of vanadium salt and oxalic acid in minimum volume of suitable solvent and was stirred for about 15 min on a magnetic stirrer. The precipitate is obtained at 5-6 pH and is washed with cold distilled water. Finally, the precipitate is washed repeatedly with dry acetone and then dried under vacuum [7].

Preparation of Vanadium Oxide (V_2O_3) : The prepared precursor is mixed with polyvinyl alcohol in the weight ratio 1:5 [8] and ground well using pestle and mortar. The resultant mixture was transferred into a crucible and ignited in an electrical oven. The dispersed phase ignited with the evolution of large volume of gases. Here, PVA reacts with the precursor a partially decomposed product was obtained, after the complete evolution of gases. The temperature of the process does not exceed 300^{o}C at any time. Chemical and physical characterisation of the partially decomposed products did not give any confirmable phases. The possible reason for a partially decomposed product formed may be attributed to the low temperature of the reaction giving rise to the insufficient energy needed for complete conversion. Hence, the partially decomposed sample mixture is further heated to get the desired product. Initially it burns with small flame and further heating the sample forms crystalline vanadium oxide (V_2O_3) product.

Density evaluation from X-ray data : The X-ray density of the samples have been computed from the values of lattice parameters using the formula [9-10].

$$d := 8\,\frac{M}{Na^3}$$

Where 8 represents the number of molecules in a unit cell of a spinal lattice
M = Molecular weight of the sample N = Avogadro's number
a = Lattice parameter of the sample
The lattice constant for the cubic was calculated using the equation

$$d = \frac{a}{(h^2+k^2+l^2)^{1/2}}$$

Tap density: Synthesized vanadium oxide was crushed in agate mortar using a pestle and mortar. A known amount of this powder was filled into a graduated cylinder of 25ml capacity. The cylinder was tapped until the powder level remains unchanged. The volume occupied by the powder was noted. The ratio between the weight of the substance and the volume gave tap density [11].

Powder density: The powder densities were measured using Archimedes principle [12] with a pycnometer and xylene as a liquid medium. The pycnometer of volume 25ml was used. The following weights were taken and used in the density calculation.

$$\rho_{sample} = \frac{(w_2 - w_1)\,\rho_{sol}}{(w_4 - w_3) + (w_2 - w_1)}$$

Weight of the bottle = W_1g, Weight of the bottle + Substance = W_2g, Weight of the bottle + Substance + Xylene = W_3g, Weight of the bottle + Xylene = W_4g, Density of Xylene = ρ_{sol}, Density of sample = ρ_{sample}

Crystallite size from X-ray data : Detailed knowledge of crystallite size, shape and strain in a finely divided powder often helps to correlate many physical properties of a system undergoing transformation in a solid-state reaction. X-ray line broadening analysis provides a method of finding bulk average size of coherently diffracting domains and r.m.s strain. The average crystallite size (D) from X-ray line broadening has been calculated using the Scherrer equation [13-14]. The instrumental broading was corrected using quartz as a internal standard.

$$D = \frac{0.9\lambda}{\beta_{1/2}\text{Cos}\theta}$$

Where λ is the wavelength of the X-ray beam, $\beta_{1/2}$ is the angular width at the half-maximum intensity and θ is the Bragg angular

Characterisation: The X-ray diffraction patterns were obtained employing a Geol JDX-8p spectrometer using CuKα radiation. The X-rays generator was operated at 30kV and 20mA. The scanning range, $2\theta/\theta$ were selected. The scanning speed $=1^0$ min^{-1} were employed for precise lattice parameter determination. High purity silicon powder was used as an internal standard. The shape, size and distribution of the powder, as prepared tin oxide sample, microstructure of the sample have examined using a Leica-440 Cambridge Stereoscan, scanning electron microscope image. The infrared spectra of the oxide sample were recorded on a Perkin –Elmer FTIR spectrophotometer [Model 1000] in the range 400 cm^{-1} to 4000cm^{-1}.

RESULTS AND DISCUSSION

X-ray diffraction : The structure of as prepared vanadium oxide is studied by X-ray diffraction tool. XRD pattern of as prepared vanadium oxide is shown in figure-1. The pattern shows large number of peaks confirms the formation of crystalline oxide material. The d-spacing values of the sample matches well with standard 44-0253 JCPDS file. Unit cell parameters ware obtained by least–square refinement of the powder XRD data. This study reveals that the sample is monophonic with high surface area.

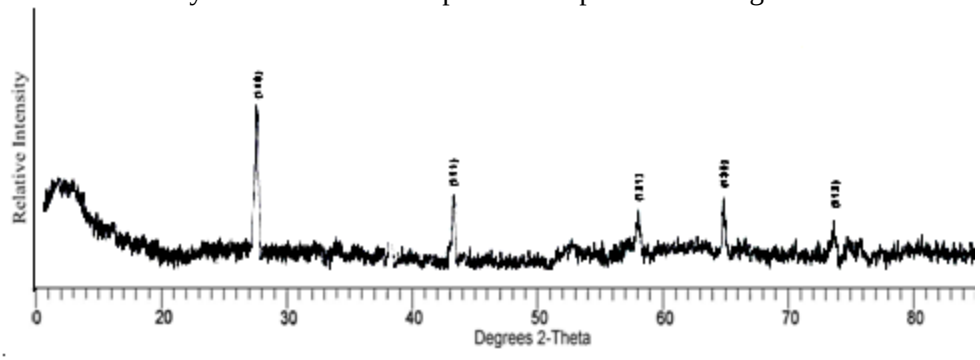

Figure1: XRD pattern of Vanadium oxide

Crystallite size and density: The crystallite size of the co sample is calculated from XRD data is 75nm. The size obtained is dependent on solid-state transformation reaction, which generally adopts the habit of its precursor. Thus, the conversion of vanadium oxalate precursor into its oxide is considered being topotactic in nature, indicating that the synthesis of precursor with very small particle sizes would be required for obtaining nanosized vanadium oxide sample. The densities of the sample calculated from XRD data, tap density and powder density is 5110 kg m^{-3}, 5089 kg m^{-3} and 3840 kg m^{-3} respectively. The sample shows approximately same density may be attributed to their average shape which might have similar surface area.

Scanning Electron Microscopy: The morphology of as prepared cadmium oxide was studied by Scanning Electron Microscope tool. Figure 2 shows SEM image of as prepared vanadium oxide sample. Particles are irregular shape and are closely arranged. Close packing with submersing particles together. Chemical homogeneity with uniform morphology and is also observed in the image.

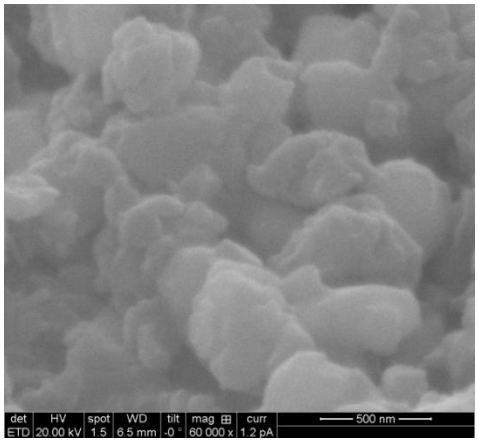

Figure 2: SEM image of Vanadium oxide

Infrared studies: The prepared vanadium oxide sample was further characterised by Fourier transform infra red(FTIR) tool. Figure 3 shows FTIR spectrum of as prepared vanadium oxide sample. The sample shows the peak around 3600cm-1 corresponds to water of absorption and the peak at 1400 cm-1 due to the presence of some overtones. The peaks 1000cm-1 corresponds to vanadium-oxygen (V-O) vibrational modes arising from inter-atomic vibrations of the spinal compound [15]).This conform the formation of vanadium oxide sample.

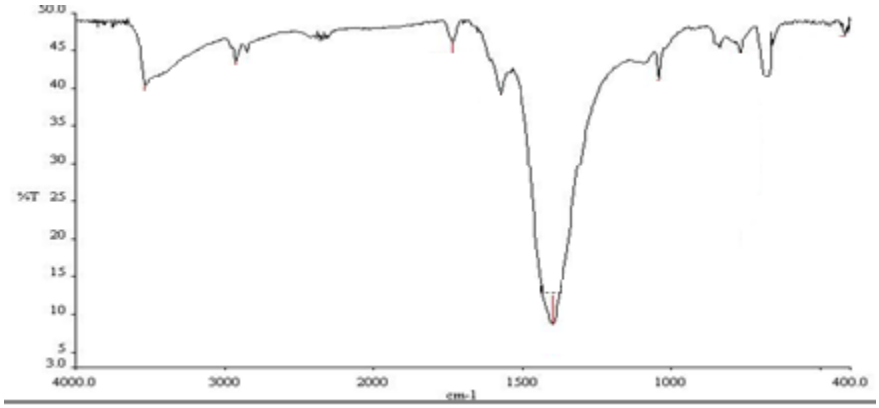

Figure 3: FTIR spectrum of as prepared Vanadium oxide sample

APPLICATIONS

This method can be applicable for the synthesis of different phased oxides at nano dimensions.

CONCLUSIONS

This method shows the solid state chemical conversion of vanadium carboxylate precursor in to its oxide nano material. The efficient polymer fuel is used in the combustion process. This preparative technique is very simple and energy efficient to obtain materials at nano dimension. Hence, this method can adopt for the synthesis of different phased oxides at nano dimensions. Vanadium carboxylate is completely converted in to its oxide, which was confirmed by characterization tools.

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