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Research Article

Calcinations Temperature Oriented Structural and Magnetic Properties of $\text{SnO}_{2\pm\mu}$ Nano-Crystalline

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ABSTRACT

At present times, the Nano-science is considered as the main focus among the today researchers/analysts because of its extreme and excellent property and reduced dimensions effectual superior application in numerous areas of science and technology. The current work emphasized the Synthesis of SnO_2 nano stuffs via microwave assisted chemical co-precipitation techniques. The samples have been calcined at temperature 200°C , 400°C , 600°C and 800°C for 2 hours in muffle furnace and obtained powdered form samples through grinded in agate mortar. The structural analysis and compositional investigations of resultant calcined samples have been investigated through the diffraction of X-Rays, imaging through electron microscopy such as FESEM and magnetic properties were accessed via VSM tools whereas, IR spectroscopy was used to functional group present in the samples. The various outcomes of the X-Ray diffraction patterns revealed about the samples possess crystalline nature of tetragonal structure geometry, the average grain size obtained by adopting the Debye Scherrer relation and was 12.66 nm , 14.50 nm and 16.07 nm and 20.82 nm for and for SnO_2 nano samples calcined at temperature 200°C , 400°C , 600°C and 800°C for 2 hours respectively. The IR spectroscopic peak at wavenumber position 2996 cm^{-1} confirmed the vibrations of water molecules which were present in atmosphere air of samples and peak at position 1086 cm^{-1} indicate the presence of carbonate residues whereas additional peaks at positions nearly 615 cm^{-1} for samples calcined at 400°C and beyond attributed O-Sn-O stretching and confirmed the formation of SnO_2 however, the solder peak at observed at 575 cm^{-1} for samples calcined at 200°C assigned to HO-Sn-OH stretching vibrations of $\text{Sn}(\text{OH})_2$ molecules. The perusals of FESEM Images of SnO_2 nano crystallites shows that the formed nano size grains possess uniformity in size, cluttered in structure and truncated spherically in form. VSM data reflects that the samples formed are ferromagnetic in nature and novel calcined samples gradually enhanced their magnetic characteristics with rise of calcination temperature upto $600^\circ\text{C}/2\text{hrs}$ and then losses magnetic characteristics for sample calcined at $600^\circ\text{C}/2\text{hrs}$.

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KEYWORDS: SnO_2 nano-crystalline, XRD, FTIR, FESEM.

1. INTRODUCTION

In the present work, SnO₂ nanomaterials were synthesized using a microwave-assisted chemical co-precipitation method, which offers advantages such as rapid reaction rates, uniform heating, and controlled particle growth. The as-prepared samples were calcined at temperatures of 200°C, 400°C, 600°C, and 800°C for 2 hr in a muffle furnace, followed by grinding in an agate mortar to obtain fine powdered samples. The influence of calcination temperature on the structural, morphological, magnetic, and vibrational properties of SnO₂ nanomaterials was systematically investigated. The Structural and compositional characterizations of the calcined samples were carried out using X-ray diffraction (XRD), IR spectroscopy and Imaging through FESEM tools and magnetic behaviour of samples summarised through VSM tools and results were discussed in upcoming paragraphs of this paper.

2. Experimental

2.1 Method of Preparation

The authors declared that all the chemicals used in this work were analytical in grade and not be further purified at laboratory scale. The various samples of Pure SnO₂NP's were synthesized via microwave irradiated chemical co-precipitation protocol with addition of Alovera extract by taking hydrated salts of SnCl₂.5H₂O(s). The appropriate concentration of SnCl₂.5H₂O was added in 100 ml doubly deionized water (DDW) along with 10 ml fresh Alovera planted extract. So white color spongy solutions were made and add 2.0 M NaOH solution (pH 14.0) of concentration was added drop wise so that pH achieved at value 9.0. The color of solution changes from white to dull white with rise of pH with addition of NaOH solution and precipitates were achieved at pH value 9.0. The precipitated

solutions were kept for ageing process about 24 hrs. and then filtered with Hoffman filter paper and thereafter, washed multiply with ethanol and DDW solution. The resulted filtered cake was given two shifts of duration 15 minutes in domestic microwave oven and then distributed in three silica crucibles for heat treatment at temperature 200°C, 400°C, 600°C & 800°C for constant heating of 2hours respectively. The calcined samples were crushed in agate mortar until resulted fine powders and then stored in vacuum bottle for further characterization techniques.

1. Instrumentation Used for Characterizations of samples

The influence of calcination temperature on the crystalline structure, magnetic properties has been studied. Structural and crystalline properties of calcined samples were examined through X-ray diffractometer (XRD) with wavelength=1.5406 Å radiation with diffraction angle in the range of $2\theta=10^{\circ}$ -80°, Elemental group was studied by Fourier Transform Infrared Spectroscopy (FTIR) with wave no. in the range of 400 cm⁻¹ to 4000 cm⁻¹.Magnetic behavior of samples was analyzed by Vibrating Sample Magnetometer (VSM) M_H Vs. H(±3.0 T).The results of above studies are given in next section of paper.

3.1 RESULTS AND DISCUSSION

3.1.1 XRD data analysis

X-ray diffraction is called finger print of materials and X-ray diffraction is an indispensable tool for the characterization of materials. It is used to study the crystalline behavior of powered samples. It is also used for the identification of the lattice structure. This method can be used for phase identification structural analysis, grain and unit cell determination.

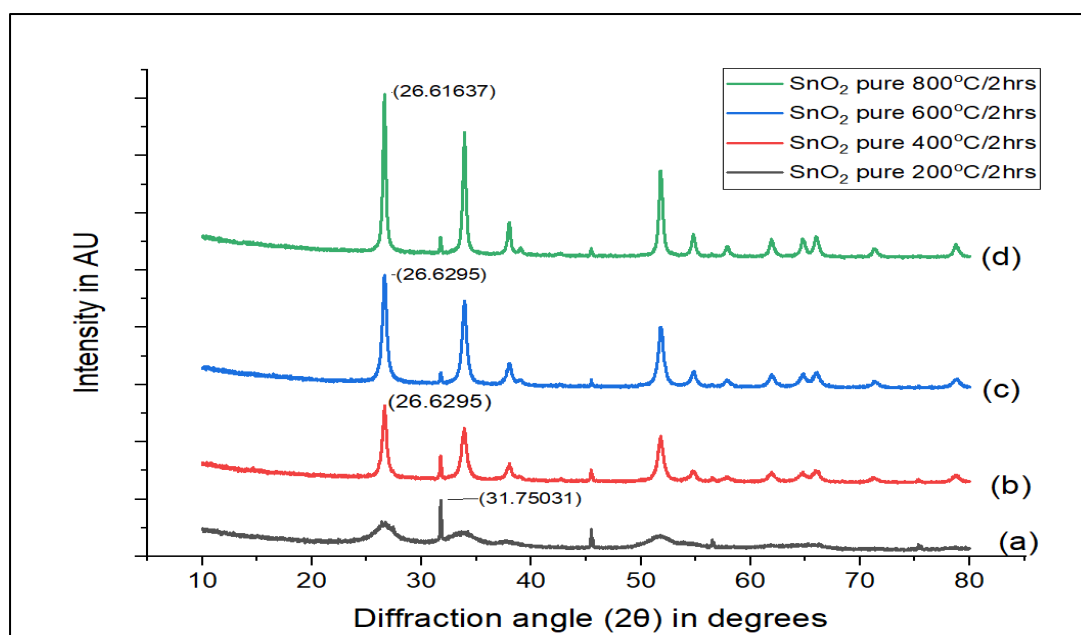


Figure 1: XRD Spectrum of SnO₂ Pure nano-crystalline with different calcination temperature at (a) 200°C (b) 400°C (c) 600°C (d) 800°C for 2Hrs

The above figure recorded X-ray diffraction (XRD) patterns for the SnO₂ nanoparticle samples calcined at 200°C, 400°C, 600°C, and 800°C clearly reveal the crystalline nature of the synthesized materials. The most intense peaks shows abnormal behaviour for sample calcined at 200°C/2hrs and positioned at $2\theta=31.7503^\circ$ and indicates the formation of Sn(OH)₂ at such small temperature and same was verified further with corresponding IR peaks whereas, the most intense peaks for SnO₂ samples calcined at 400°C, 600°C and 800°C for 2hrs were more or less similar at position $2\theta=26.6295^\circ$ also the

observed diffraction peaks are in good agreement with standard JCPDS card No. 41-1445 confirming the formation of single-phase tetragonal rutile SnO₂. (rutile SnO₂), indicating phase purity and structural stability of the samples. The increase in X-ray diffraction peak intensity with rising temperature confirms the improvement in crystallinity of the material. At higher calcination temperatures i.e. 600°C atoms acquire sufficient thermal energy to rearrange into more ordered lattice positions resulting in a better-defined crystal structure.

Table 1- XRD data of SnO₂PureNanocrystallines calcined at different temperatures for 2 hrs

Sr No.	Pure SnO ₂ Samples calcined at different temperature for 2hrs	Peak position(2θ) (In degree)	FWHM (In degree)	Size (in nm)
1.	200°C	31.7503	0.6528	12.65
2.	400°C	26.6295	0.5631	14.50
3.	600°C	26.6295	0.5532	16.07
4.	800°C	26.6163	0.3715	20.82

The above XRD table analysis shows that FWHM of the diffraction peaks decreases with increasing temperature, suggesting sharper and more well-defined peaks and the thermal treatment also reduces lattice defects, dislocations and macro-strains improving crystalline aspects. Consequently, the average crystallite size increases with calcinations temperature from 12.65 nm to 20.82 nm. The combination of stable peak intensity, decreasing FWHM, and increasing size reflects the structural stability and temperature-driven grain growth of SnO₂. Overall, calcination improves crystallinity while promoting the growth of larger, defect-free nanoparticles.

3.1.2 IR spectroscopic results analysis

The Fourier Transform Infrared Spectroscopic tools provides

direct information about the chemical composition, bonding, and molecular interactions in a material. Therefore, the characterization of these powders cannot be completed without the identification of the surface chemical species. In present work, IR region 400 cm⁻¹- 4000 cm⁻¹ is used to analyze the functional vibrational group present in various samples.

Figure shows the FTIR spectrum of the synthesized SnO₂ sample. An absorption band observed at 2996 cm⁻¹ is attributed to the stretching vibrations of adsorbed water molecules present on the sample surface, indicating moisture adsorption due to the high surface area of the nanoparticles. The band appearing at 1086 cm⁻¹ corresponds to carbonate residues, which may originate from atmospheric CO₂ adsorption or incomplete removal of precursor species during synthesis.

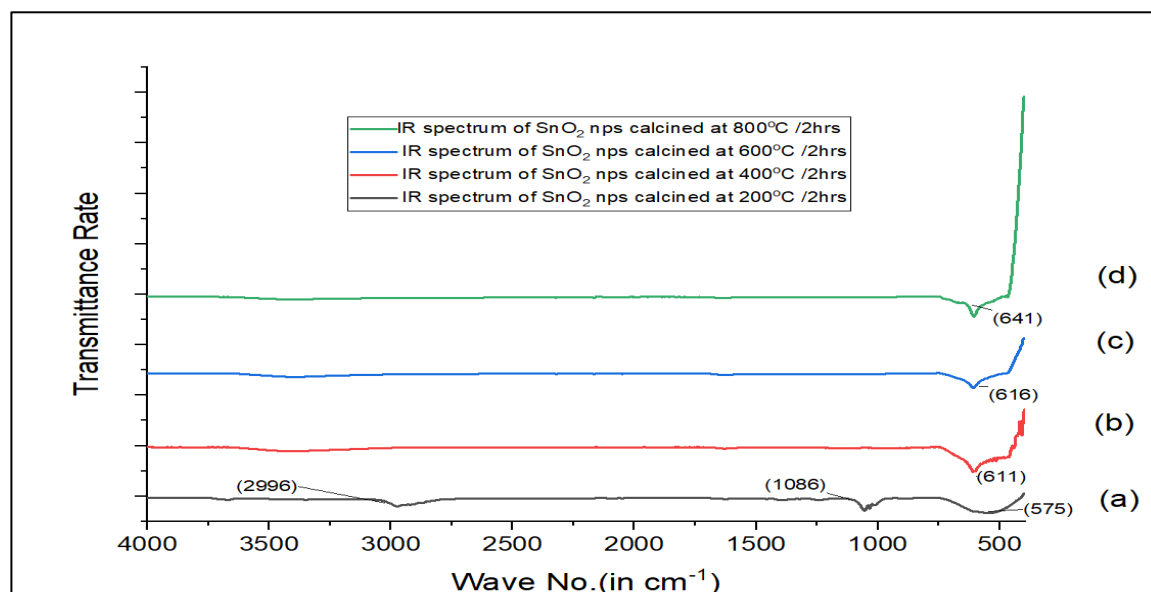


Figure 2: IR Spectrum of SnO₂-nano-crystalline calcined for 2 hrs at different temperatures at (a) 200°C (b) 400°C (c) 600°C (d) 800°C for 2Hrs

The prominent absorption sharp peak at 611 cm^{-1} , 618 cm^{-1} and 641 cm^{-1} were assigned to O–Sn–O stretching vibrations and confirmed the formation of crystalline SnO_2 at temperature $400^\circ\text{C}/2\text{hrs}$ and beyond whereas, the peak positioned at 575

cm^{-1} for sample calcined at $200^\circ\text{C}/2\text{hrs}$ were corresponds to HO–Sn–OH vibrations of $\text{Sn}(\text{OH})_2$ molecules respectively. The IR results support the candidature of researcher for synthesis of SnO_2 nano-crystalline.

3.3 FESEM

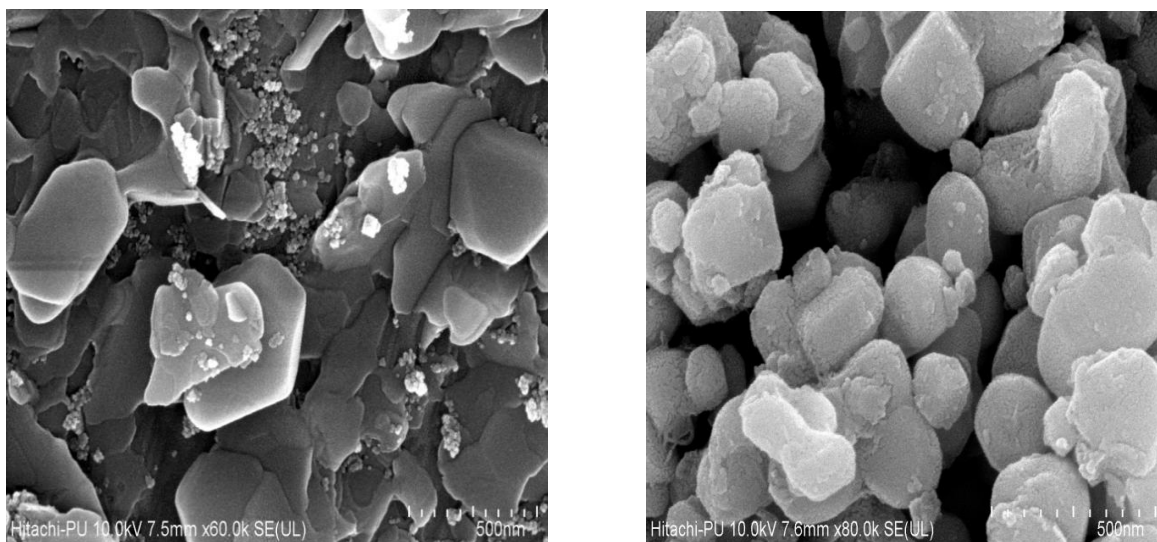


Figure 3: FESEM Image of pure SnO_2 nano-crystalline calcined at temperatures (a) $600^\circ\text{C}/2\text{ hrs}$ (b) $800^\circ\text{C}/2\text{ hrs}$

The perusal of image shows that 2D hexagonal shape were observed for sample SnO_2 calcined at $600^\circ\text{C}/2\text{hrs}$ or 2D sheets were in information with thickness few nm and lengths are in about 200 nm whereas for sample SnO_2 calcined at $400^\circ\text{C}/2\text{hrs}$ are 3D stone like crystals information at nano scale. Moreover, the exhibitance shows that polycrystalline were in formation which were well dispersed and sharp transmittance.

1.1.4 M-H graph results analysis

The Vibrating sample magnetometer is a device used to measure magnetic properties of materials. It works on Faraday's Law of Electromagnetic Induction where vibration of a sample in a magnetic field induces voltage. The induced signal is proportional to the sample's magnetization. It provides the M-H curve showing properties like coercivity and saturation magnetizations is widely used in nanomaterials and magnetic materials research. It is fast, sensitive and suitable for small samples.

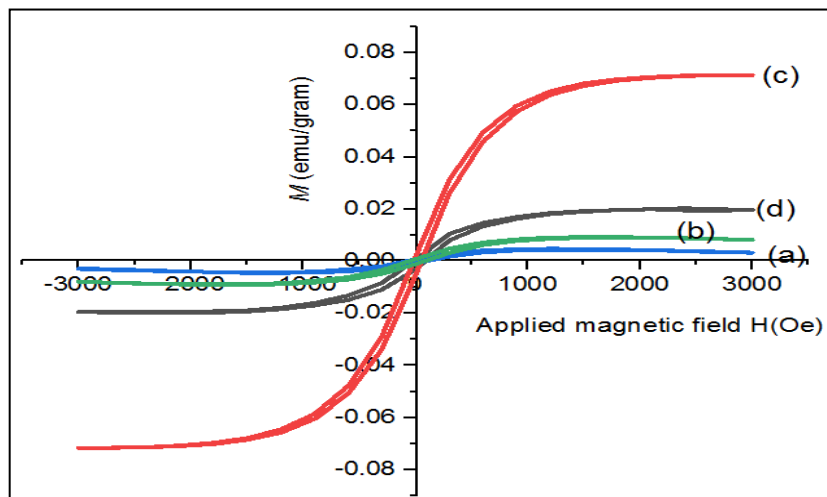


Figure 4: M-H curves of pure SnO_2 Nps calcined at temperature (a) $200^\circ\text{C}/2\text{ hrs}$ (b) $400^\circ\text{C}/2\text{ hrs}$ (c) $600^\circ\text{C}/2\text{ hrs}$ (d) $800^\circ\text{C}/2\text{ hrs}$ respectively.

The perusal of above M-H graph shows that the newer calcined samples enhanced their magnetic properties with smaller rate with rise of calcination temperature from calcination temperature 200°C/2hrs to 400°C/2hrs thereafter, the saturation intensity of magnetization have optimum enhancement at calcination temperature 600°C/2hrs. whereas, further increase of calcination temperature the sapeciman losses there magnetic behaviour. The SnO₂ nano-particulates calcined at 600°C/2hrs were superior magnetic properties and pronounced applications in the area of MRI applications, spintronic and fabrication of memory chips.

4. CONCLUSIONS

The various outcomes of the X-Ray diffraction patterns revealed about the samples possess rutile tetragonal structure geometry and the average grain size obtained by adopting the Debye Scherrer relation and was to be 12.66 nm, 14.50 nm and 16.07 nm and 20.82 nm for and for SnO₂ nano samples calcined at temperature 200°C, 400°C, 600°C and 800°C for 2 hours respectively. The IR spectroscopic peak peaks at positions 615 cm⁻¹ for samples calcined at 400°C and beyond attributed O-Sn-O stretching and confirmed the formation of SnO₂ however, the solder peak at observed at 575 cm⁻¹ for samples calcined at 200°C assigned to HO-Sn-OH stretching vibrations and confirmed the formation of Sn (OH)₂ molecules. The FESEM Images shows that the formed nano size grains possess uniformity in size, cluttered in structure and truncated spherically in form. VSM data reflects that the samples formed are ferromagnetic in nature and novel calcined samples gradually enhanced their magnetic characteristics with rise of calcination temperature upto 600°C/2hrs and then losses magnetic characteristics for sample calcined at 600°C/2hrs. The Novel sample calcined at 600°C/2hrs was proving better magnetic properties and might be the pronounced applications in the area of MRI applications, spintronic and fabrication of memory chips.

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