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Variation in Structural and Magnetic Properties with Calcination Temperature in $Snzno_{3\pm\Delta}$ Nano-Crystallines at Fixed Zn Concentration

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ABSTRACT

Since last three decades, science and technology of particles are proving exponential growth at nano scale because of its highlighted properties and applications in respective fields. The metal oxide nanomaterials have colourful properties because of multivalency d and s subshell electronic configurations. In present work, the researcher elaborates the conclusive properties of SnO_2 and Zn doped SnO_2 nano-composites and thereafter, exploded her study with the rise of calcination temperature.The duration of calcination was opted as 2 hours because of constant heating or uniformity were occurred in about 2 hours and the temperature was risen limited up to 800°C because of met allistic particle such as SnO_2 or nano composite lost their magnetic behaviour above 800°C and beyond. The graphical representation of X-Ray diffraction data of pure SnO_2 nanoparticles (Np's) and Zn doped SnO_2 all calcined samples were more or less same in position and reflects that Zn2+ were well incorporated in SnO2 tetragonal Sn ion sites. However, the crystallite size increases with rise of calcination temperature. The IR peaks occurred at position 612cm-1, 493 cm-1 and 1048cm-1 were assigned to O-Sn-O, O-Zn-O and Sn-OH/ Zn-OH molecular vibrations. The Magnetic behaviour of all calcined samples reflects that noval materials were ferromagnetic in nature and magnetic properties were rises with calcination temperatures.

Keywords: ZnO/SnO_2 nanocomposites, XRD, FTIR, VSM tecniques.

INTRODUCTION

Tin and its compounds, such as tin oxide (SnO_2) and tin chalcogenides (Sn-S,Sn-Se) have attracted significant research interest due to their semiconducting nature, tuneable band gaps and excellent chemical stability. These nanomaterials exhibit diverse applications in energy storage devices, gas sensors, photocatalysis and optoelectronics. In particular, SnO_2 nanoparticles demonstrate high sensitivity toward gases like CO, $H_2$and NO_2 while their nanoscale structure enhances surface reactivity and sensing performance. Tin-based materials also serve as efficient anodes in lithium and sodium-ion batteries, offering high capacity and improved cycle stability.

Moreover, their notable photocatalytic activity under UV–visible light, environmental compatibility and ease of synthesis make them promising candidates for sustainable nanotechnology applications.Zinc-based nanomaterialsparticularly ZnO nanoparticles are widely used in nanotechnology due to their semiconducting, photocatalytic and antimicrobial properties.Their large surface area, stability and biocompatibility make them suitable for sensors, solar cells, and biomedical applications.Moreover, ZnO is an eco-friendly and cost-effective material for advanced nanotechnological research.

2. Experimental

2.1 Method of Preparation

The authors declared that all the samples were analytical in grade and not be purified at laboratory scale. The various samples of Pure SnO_2NP's and Zn doped SnO_2NCs were synthesized via microwave irradiated chemical co-precipitation protocol using Alovera extract by taking hydrated salts of $SnCl_2.5H_2O$ and $ZnCl_2.5H_2O$. The appropriate concentration of $SnCl_2.5H_2O$/ $ZnCl_2.5H_2O$ 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 approx. 9.0.

The color and pH changes with addition of NaOH solution and precipitates were achieved at pH value approx. 9.0.The precipitated solutions were kept for ageing process about 24 hrs. filtered with Hoffman filter paper and thereafter, washed multiply with ethanol solution. The resulted filtered cake was given two shifts of duration 15 minutes in microwave oven and then distributed in three silica crucibles and heated for 2 hours in furnace at 200°C, 400°C, 600°C & 800°C respectively. The calcined samples were crushed in agate mortar until resulted fine powders and then stored in vacuum bottle for further characterization techniques.

3. Characterization

3.1 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 and for the identification of the lattice structure. This method can be used for phase identification structural analysis, grain and unit cell determination. The calculation for inter planar spacing $d_{(hkl)}$ values is performed by Braggs law. The sample XRD is identifying with JCPDS file. The X-ray diffraction peaks of Zn doped SnO_2 have been done by Perkin Elmer diffractometer at CIL, LPU, Chandigarh over the wide-angle range 2θ =10° to 80°.The comparative analysis of XRD data of various calcined Zn doped SnO2 ncs were represented in figure no.1 as under:

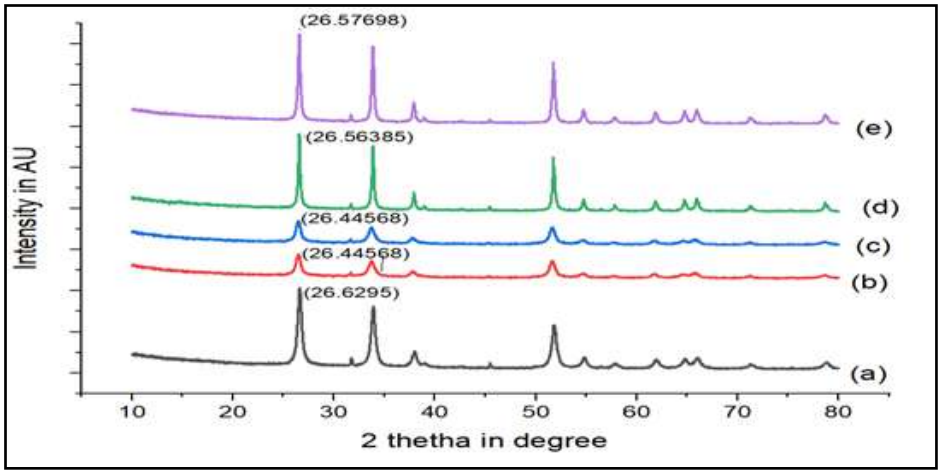

Figure 1: XRD Spectrum of SnO_2-ZnO nanocrystalline with Zn concentration 10% calcined for 2 hrs at different temperatures (a) pure SnO_2 at 600°C, (b) at 200°C, (c) at 400°C (d)at 600°C, (e) at 800°C respectively

Figure shows the XRD patterns of the SnO_2/ZnO.With rise of calcination temperature, the intensity of most intense peak rise with temperature which might be due to better oxidation took place at higher calcination temperature 200°C, 400°C, 600°C and 800°C respectively. Hencefourth, the graphical representation of SnO_2 nanoparticles were well matched with JC PDF file number JCPDS41-1445and conferming the formation ofsingle phase tetragonal routile type structure of $SnO_2$with good crystallinity where as Sn ion position at centralised and oxygen took place at corner position of molecules.In addition, the peaks at 2θ = 26.3°, 33.6°, 37.9° andthe diffraction patterns of the synthesized ZnO NPs match well with the standard Bragg positions for the hexagonal phase of wurtzite ZnO (JCPDS 04 -0831) [14-16] and confirms the formation of pure metallic zinc and hexagonal close packed crystal structure.

Table 1- XRD data of ZnO Nano crytalline materials calcined for 2 h and at temperature 200°C,400°C,600°C & 800°C

Sr No.	Sample	Peak position(2θ) (In degree)	FWHM (in degree)	Size (in nm)
1.	SnO_2 pure(600°C)	26.63	0.55	14.76
2.	SnO_2 with 10% Zn doping(200°C)	26.44	0.42	17.86
3.	SnO_2 with 10% Zn doping(400°C)	26.44	0.38	21.37
4.	SnO_2 with 10% Zn doping(600°C)	26.56	0.35	23.39
5.	SnO_2 with 10% Zn doping(800°C)	26.58	0.33	24.47

The above tabular data presented that intensity of most intense peak gradually increases with rise in calcination temperature and FWHM decreases with rise of temperature or FWHM broaden or contractionwhereas crystallite size increases with rise of temperature which might be due to better oxidation state took place at higher temperatures or O-Sn-O lattice energy increases with increase in temperature lattice energy or vibrational energy.The perusal of above XRD graph or the graphical representation of X-Ray diffraction data shows that there is no face transformation occurred with addition of Zn concentration in SnO_2 structure that is no significant changes were notice in most intense peak of various Zn doped SnO_2 nanocomposites.

FTIR data analysis

The characterization of nano powders cannot be completed without the identification of the surface chemical species. The inorganic nanomaterials are often surface with organic molecules or sometimes composites with organic material.The functional vibrational group present in various samples were analysed by scanning of samples in IR region 400 cm^{-1}-4000 cm^{-1}. The absorption pattern is helpful tool and provide finger print of samples the IR spectrum of samples. Furthermore, the individual peaks were analysed via Deconvolution of IR graph of various samples and shown in graphical representation as below in figure number 3:

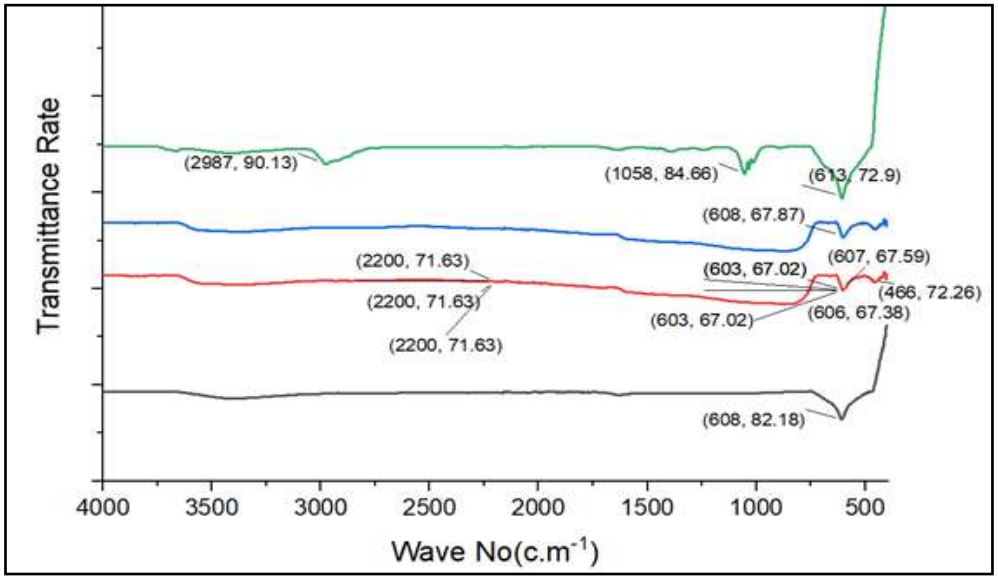

Figure3: IR Spectrum of SnO_2-ZnO nanocrystalline with Zn concentration 10% calcined for 2 hrs at different temperatures (a) pure SnO_2 at 600°C, (b) at 200°C, (c) at 400°C (d)at 800°C respectively.

The IR spectrum of various calcined samples shows that as usual broad band were found in all the graphs at about 3400 cm^{-1}, 3000 $cm^{-1}$and 1600 cm^{-1} respectively and indicates the presence of O-H vibrations of water molecules present in the atmosphere of samples. Moreover, metal oxide peaks were observed at about 608 cm^{-1},603 cm^{-1}, 566 cm^{-1}, 561 cm^{-1} and 477 cm^{-1} respectively. The peak exhibited at position 608 cm^{-1} may be assigned by O-Sn-O molecule vibration whereas peak positioned at 477 cm^{-1} be assigned to O-Zn-O vibrations of ZnO molecules. The study shows that a newer peak positioned at 562 cm^{-1} and 567 cm^{-1} were might be due to O-Zn-O-Sn-O vibrations of SnO_2-ZnO NCs. The IR spectrum results of samples were in accordance with XRD results and proved the candidature of researcher.

***M-H* data analysis**

The pure SnO_2 calcined at 600 degree Celsius / 2 hours and Zn (10%) doped SnO_2 nanocomposite samples were scanned over a wide external range of 1.5 Tesla. The various magnetic properties were attributed by these samples.

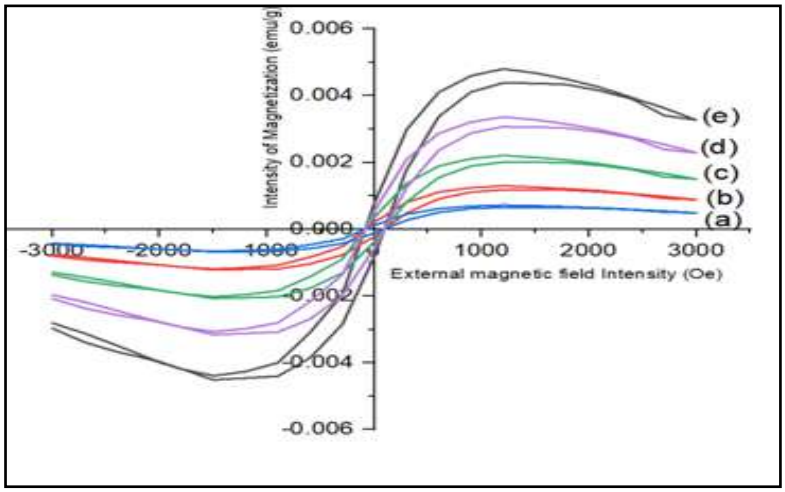

Figure 3: M-H curves of (a) SnO_2pure and SnO_2-ZnO(10%)samples calcined at (b) 200°C (c) 400°C (d)600°C (e) 800°C for fixed duration2 hrs respectively.

The aforementioned M-H curve reflects that all the samples were ferromagnetic in nature at nano-scale with different scale of M and H values. However, the ferromagnetic character rise with rise of calcination temperatures

Table 2-The Hysteresis parametric study of Zn doped SnO_2 nanocomposites calcined at different temperatures:

Sr No.	Sample calcined at temp	Ms. Saturation (emu/g)	Coercive (Hc Oe)	Remnant (emu/g)	Squareness Factor	M at Max Field (emu/g)	Energy Loss(Oe)	Saturation Applied H_s(Oe)
1.	200°C	315.222E-6	81.965	102.069E-6	1.321	261.809E-6	254.897E-3	498.30
2.	400°C	610.211 E-6	125.671	170.451 E-6	0.654	468.004 E-6	578.96E-3	639.42
3.	600°C	1.067E-3	149.590	307.781E-6	0.336	1.015E-3	1.025E+0	968.70
4.	800°C	7.858E-3	55.183	1.132E-3	0.325	7.318E-3	1.544E+0	1127.57

A comparison of the magnetic characteristics of 10% Zn-doped SnO_2 nanoparticulates calcined at various calcination temperatures for duration of 2 hours was done using Table 2. The results showed that retentivity, corresivity and saturation field rose at 800°C .Additionally, the maximum energy loss demonstrated increasing behavior as the temperature increased. The disorder in spin angular momenta of the Zn^{2+} ion with increasing temperature may be the cause of the variance in magnetic properties of different calcined samples. The study's findings show that the more recent materials have qualities that are advised for the stable creation of a transformer's Iron core within the appropriate temperature ranges.

CONCLUSION

The pure SnO_2 and ZnO doped SnO_2 samples were successfully prepared via microwave irradiated chemical co-precipitation method and thereafter, the powdered samples were calcined at 200^{o}C to 800^{o}C for2 hours. The various calcined samples have been analyzed with X-Ray diffraction and data reflects that parental SnO_2 single phase tetragonal rutile structure was conformed. Moreover, the crystallite size increases with increase with rise of calcination temperature. The strong and sharp IR peaks supports the candidature of researcher. The VSM analysis classified the samples to be ferromagnetic in domain and magnetic character increases with increase of calcination temperature of ZnO doped SnO_2 samples. The novel calcined samples might have remarkable properties in MRI application, spintronic and in fabrications of memory chips.

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