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# To Study the Effect of Calcinations Temperature on the Structural and Magnetic Properties of $ZnMnO_{3\pm\delta}$ Nanocrystallines

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**ABSTRACT**

**In present scenario, the technological applications of nanomaterials are at key position because of unique in Physico-chemical property at this scale. The present research work explored the effect of concentration of Zn element on structural and morphological modifications in Zinc Oxide Manganese Oxide nano-crystalline. In this research work, nano-sized Zinc Oxide Manganese Oxide sample was synthesized via advanced co-precipitation chemical protocol. The specimens have been calcined at $200^0$C, $400^0$C, $600^0$C for a time span of 2hours. The XRD results reflects that The crystalline size of newer nano-crystallines were increase in particle size with increase in calcinations temperature Zinc Oxide Manganese Oxide hexagonal structure .The FTIR tools was used to examine the purity of samples and entities of Zinc Oxide Manganese Oxide structural material. The magnified images of samples were seen through FESEM and HRTEM tools and exhibited structure were 2-D nano-sheets were in formation. The M-H curve of calcined samples concluded that nature of all calcined samples were paramagnetic in nature and magnetic susceptibility shows inverse behaviour with rise of temperature and follow Curie-Weiss Law of magnetism.**

**Keywords: Zinc Oxide, Debye-Scherrer, VSM, XRD, FTIR, FESEM and HRTEM etc.**

## INTRODUCTION

The superior properties of nanomaterials are widely recognized, making them a popular topic due to their numerous enhanced applications. Researchers are highly interested in these materials because they believe many possibilities remain unexplored. Nanomaterials could lead to significant advancements, as their unique characteristics at the nanoscale such as morphology, size, and agglomeration behavior—hold great potential. These nanoscale properties enable superior performance in various fields and innovations that rely heavily on nanotechnology's unique capabilities. Nanomaterials differ significantly from their larger counterparts due to a high surface-to-volume ratio and quantum effects impacting their optical properties. At the nano-scale, it was examined that the effect of quantum channels has a huge influence on the optical features of the stuff. The ZnO nano-flecks are of immense significance. As we had seen before that the nano-scale composites of Zinc Oxide alongside Transition Metal Oxides at variant dopant concentrations showed distinct/unique properties as well as behaviour at the nano-scale.

Manganese Oxides fetched quite more attention towards itself as it possesses much significance both in technology and in science. Since, the Manganese has various stable oxidation states; it can form the various kinds of oxides. Among other metal oxides, special surveillance has been focused on the fabrication/preparation and properties of Manganese Oxide (MnO2), which is an important role in some specifically, applied usages like as in the field of liquid crystal displays, high-temperature resistant materials, corrosive resistant materials, green pigment, catalysts, etc. In this work the various samples of Zinc Oxide Manganese Oxide were synthesized via advance chemical co-precipitation protocol and the Structural analysis of composite materials were done with the help of X-ray diffraction, Fourier transform infra-red spectroscopy, VSM, FESEM and HRTEM image magnifications.

### Synthesis Techniques

The nano stuffs of ZnO doped $MnO_2$ were synthesized by the Micro-wave treated co-precipitation advance synthesis techniques. The ignited stuff of various concentrations ignited at various temperatures for fixed duration.The slurry of the appropriate concentration of $Mn(NO_3)_2.4H_2O$ (HIMEDIA, India) and $ZnCl_2.6H_2O$ (HIMEDIA, India) was foregathered/mixed in the de- ionized water of 100 ml. Then the slurry of $NH_3$ was poured in the above said slurry at

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$100^0$C temperature and the finalize amalgam was stirred at the temperature of 25 $^0$C for 2 hours of time span using magnetic stirrer and then the concluding amalgam was retained for the ageing process at room temperature for at least 24 hours. On completion of reaction, the concluding creamy-white colored precipitates were strained and then made to wash with the doubly distilled water. These precipitates were also washed then with ethanol or Merck for manifold times to get rid of the impurities or by-products. The filtered cake was then dry in the air at 100°C temperature for 4 hours of time span. The as-synthesized specimens of the different concentrations had been ignited eventually for various time scales and at a particular temperature in the air. The specimen was crushed in an agate mortar to obtain a fine powder of ZnO-doped $MnO_2$ for further characterization.

## RESULTS AND DISCUSSION

### Structural characterization:

To allocate the framework of ignited specimen by using X-ray diffractometer with copper ($CuK_\alpha$)radiation ($\lambda$ = 1.5408 Å) in the confine of $10^0$–$80^0$ the powder sample studies using X-rays have been accomplished. The XRD designs of various Zinc Oxide doped Manganese Oxide nano-sized stuff ignited at 600$^o$C temperature for 2 hours are exhibited in

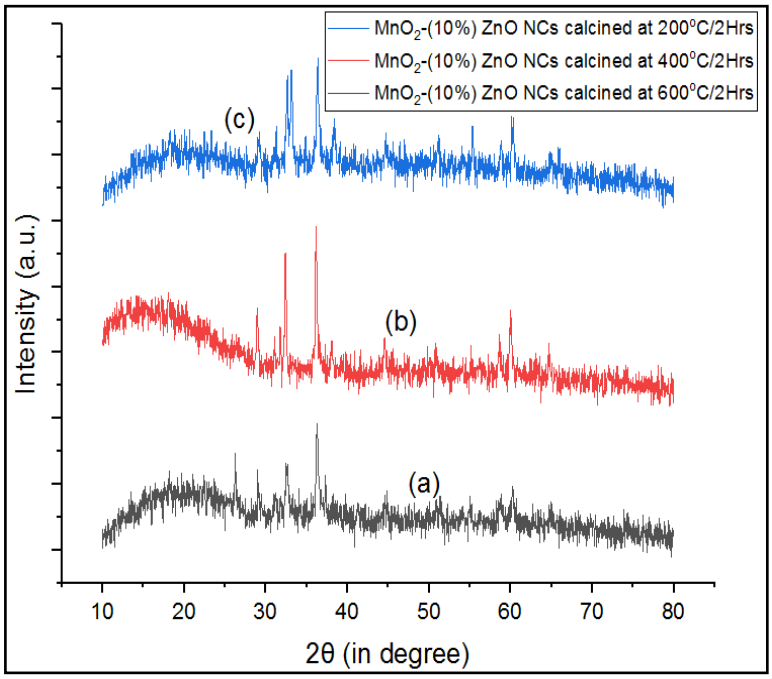


**Figure-1: Zinc Oxide doped $MnO_2$ (10%) nano-sized stuff ignited for 2 hours at 200$^o$C, 400 $^o$C, 600 $^o$C.**

The grain size for Zinc Oxide doped ZnO(10%)/ $MnO_2$ nano-sized stuff ignited at 200 $^0$C for 2 hours is 26.76 nm, Zinc Oxide doped ZnO(10%)/ $MnO_2$ nano-sized stuff ignited at 400 $^0$C for 2 hours is 37.44nm and for Zinc Oxide doped ZnO(10%)/ $MnO_2$ nano-sized stuff ignited at 600 $^0$C for 2 hours is 38.17nm. It is as Co-atom having more atomic radii measure than that of Al-atom concluding in rise in the grain size of nano-sized stuff with rise the temperature of specimens of Zinc Oxide doped $MnO_2$ nano-sized stuff.

**Table-1: XRD Zinc Oxide doped $MnO_2$ (10%) nano-sized stuff ignited for 2 hours at 200$^o$C, 400$^o$C, 600$^o$C.**

| Sr. No. | Calcinations temperature ($^o$C) | 2Θ (Degrees) | FWHM (Radians) | Crystallite size (nm) |
|---|---|---|---|---|
| 1 | 200$^o$C | 36.18 | 0.527 | 26.71 nm |
| 2 | 400$^o$C | 36.30 | 0.452 | 31.13 nm |
| 3 | 600$^o$C | 36.51 | 0.413 | 34.08 nm |

It was observed that the location/position of XRD alps are more or less same values but the intensity of XRD spectrum peaks shows increasing behavior with rise of temperature along with small decrement in β occurs with rise of temperature i.e., the value of β decreases with increase of temperature. Which implies that the size of ZnO(10%) doped $MnO_2$ nano-crystallines increases with rise of temperature. It might be due to rise of lattice energy with rise of temperature

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**FTIR Spectroscopy analysis**

The IR spectroscopic tools were used to identify the group/ contamination particles/ other entities present in the samples. The transmittance rate were noted with wave number of radiation incident on samples ranging from 400-4000 cm-1.The FTIR electromagnetic spectrum of synthesized specimen of ZnO doped $MnO_2$ nano-sized stuff ignited at $200^0$C, $400^0$C, $600^0$C for 2hrs. The IR data were represented in graphs as shown in Figure-2.

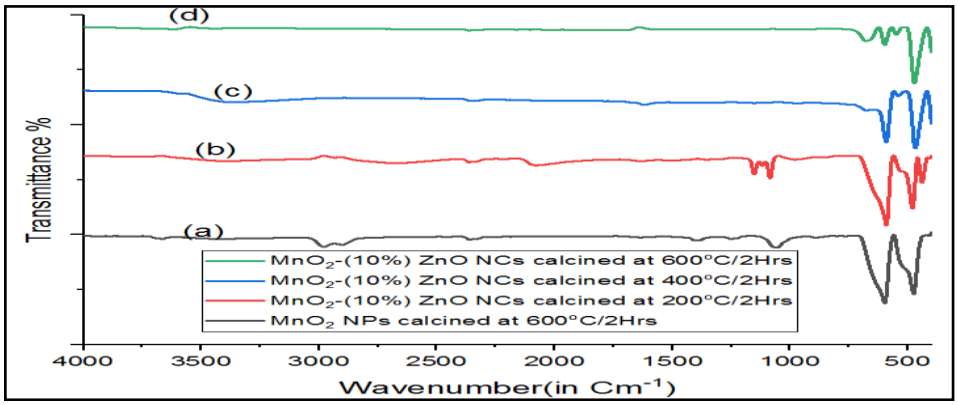


**Figure - 2: FTIR Spectra of Zinc Oxide doped $MnO_2$ (10%) nano-sized stuff ignited for 2 hours for distinct temperature (a)200°C (b)400°C (c)600 °C.**

The FTIR analysis of the synthesized Manganese Oxide nanoparticles revealed several characteristic peaks, indicating the presence of various functional groups. At peak positions 568 $cm^{-1}$ and 438$cm^{-1}$, Mn-O-Mn and Zn-O-Zn vibrations were identified. A peak at 691 $cm^{-1}$ corresponds to Zn-O-Mn bonding. Additionally, peaks at 1546 $cm^{-1}$, 2330 $cm^{-1}$, and 3402 $cm^{-1}$ indicate the presence of -OH groups. These -OH group vibrations are due to water molecules present in the atmosphere, which interact with the nanoparticles. This detailed analysis helps in understanding the chemical structure and bonding of the synthesized Manganese Oxide nanoparticles. FTIR spectra of the Zinc Oxide doped $MnO_2$ (10%) nano-composites ignited for 2 hours at 200 $^0$C, 400 $^0$C, 600 $^0$C of assembled specimen are exhibited. The perusal of the image exhibits that consign of the ignited specimens rises with rise in calcinations temperatures. It might be due to rise of the condensation of the oxygen throughout calcinations course of action.

**M-H Curve Analysis**

The magnetic properties of various samples were explored through deploying vibrating sample magnetometer tools and the applied field ranges were taken from 0-10,000 Os. In present work the various calcined samples of ZinC (Zn) 10% doped manganese Oxide nanocrystalline were studied through vsm tool and result were compare with pure manganese Oxide sampled calcined at 600°C for 2 hrs. The data received from lab CEERI Pilani were represented in various graph.

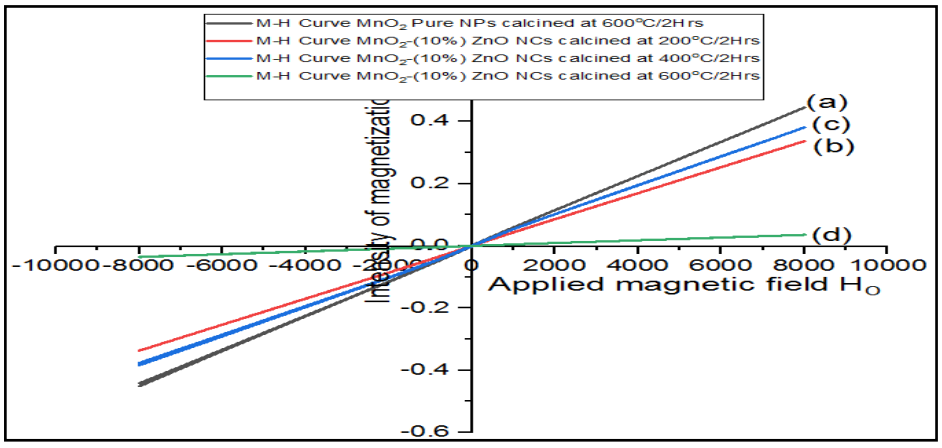


**Figure:3-M-H Curve of (a)pure $MnO_2$ calcined at 600°C/2hrs and Zn( 10%) doped $MnO_2$ specimen calcined(b)200°C/2hrs(c) 400°C/2hrs(d) 600°C/2hrs**

The perusal of above M-H-graph represents that magnetic nature of particles are paramagnetic in nature with no hysteresis loss.

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**Table 3: Magnetic data of pure $MnO_2$ calcined at 600 $^oC$/2hrs and Zn (10%) doped $MnO_2$ specimen calcined at 200$^oC$, 400$^oC$& 600$^oC$ for 2hrs.**

| Sr. No. | Sample Name | Saturation Magnetization( 10-3 emu/g) | Coercive Field (in Hc Oe) | Remanent Magnetization ($10^{-3}$ emu/g) | Squareness | Double Squareness Factor | Maximum Permeability (in $10^{-6}$ emu/g/ Oe) | Max. energy loss( in $10^{-3}$ MGsOe) | Sat. field, field (0.95 Ms) in $X10^{-6}$ Hs Oe |
|---|---|---|---|---|---|---|---|---|---|
| 1 | $MnO_2$ Pure | 447.886 | 24.97 | 1.518 | 0.003 | 0.035 | 60.947 | 447.886 | 54.924 |
| 2 | Zn(10%) doped 200$^oC$ | 336.716 | 9.574 | 0.423 | 0.991 | 0.647 | 44.074 | 336.716 | 41.958 |
| 3 | Zn(10%) doped 400$^oC$ | 382.100 | 16.880 | 0.938 | 0.002 | 0.047 | 55.876 | 382.100 | 45.626 |
| 4 | Zn(10%) doped 600$^oC$ | 35.725 | 9.946 | 46.649 | 0.001 | 0.158 | 4.717 | 35.725 | 4.399 |

The M-H curve of calcined samples concluded that nature of all calcined samples were paramagnetic in nature and magnetic susceptibility shows inverse behaviour with rise of temperature and follow Curie-Weiss Law of magnetism. The above mentioned tabular data reflects that saturation magnetization, coercive field, maximum permeability and maximum energy loss initially increase with increase of calcinations temperature up to 400$^oC$/ 2hrs thereafter, gradually decrease in saturation magnetization, maximum permeability and maximum energy loss and continuously decrease coercive field at and above calcinations temperature 600$^oC$/2hrs . It might be due to occurrence localized domain in the crystal with rise of temperature such as 600$^oC$/ 2hrs.

**HRTEM Images Analysis**

HRTEM micro-graphs of Zinc Oxide doped $MnO_2$ nano-composites with concentration 10% ignited at 600 $^0C$ for 2 hours were exhibited in figure-3**.**

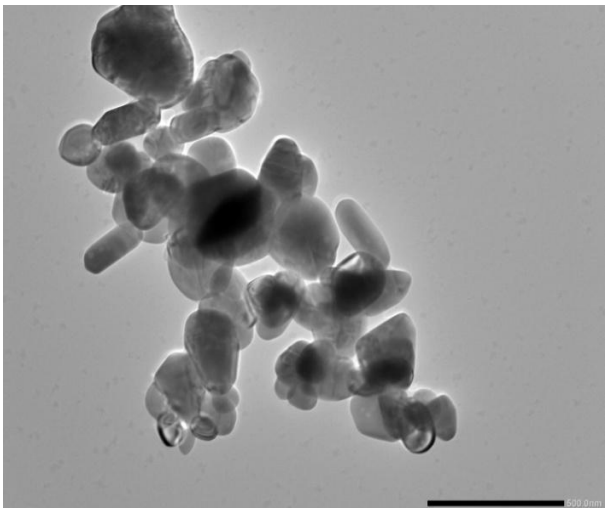

**Figure-4: HRTEM images of ZnO doped $MnO_2$(10%)nano-sized stuff ignited at 600 $^0C$ for 2 hours**

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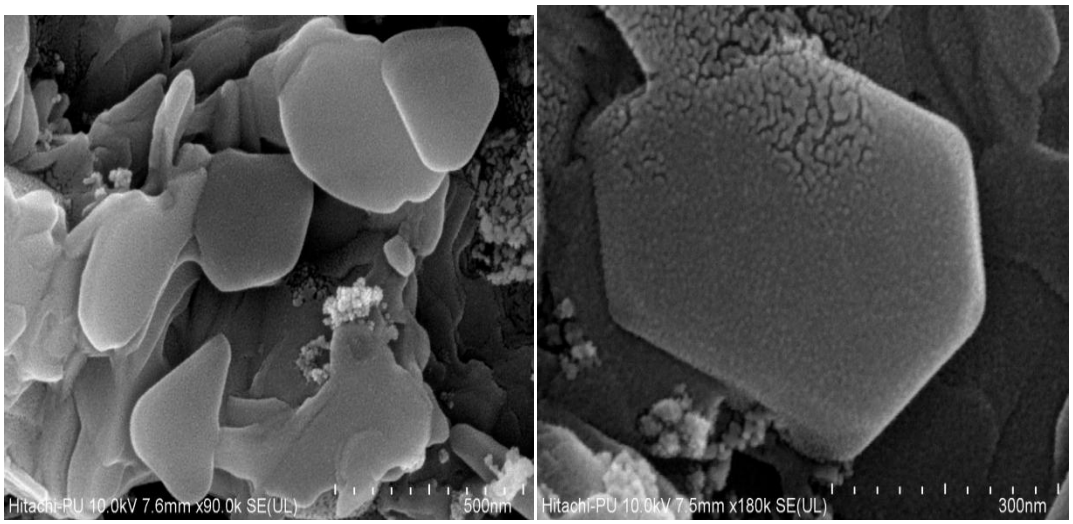
Examination of the Image exhibits that diameter of the nano-sized stuff ranging from 26 to 38 nm and intermediate grain size estimated to be 32nm. The micrographs of TEM concluded that grain size results resemble with XRD results and clarified that grain size rises with doping molar concentration. From the micro-graph, it was inspected that the nano-fleck are polycrystalline kind and spherical in contour.

**FESEM images analysis**

The scanning of sample through electron microscopy images of Zinc Oxide doped $MnO_2$ nano-sized stuff ignited at 600 $^0$C for 2 hours were more or less similar to typical scanning of sample through electron microscopy. Micrograph of Zinc Oxide doped $MnO_2$ (10%) nano-composites ignited at 600$^0$C for 2 hours is exhibited in Figure-4**.**




**(a)** **(b)**

**Figure -5: FESEM images of ZnO (10%) doped $MnO_2$ nano-sized stuff ignited for 2 hours (a)400$^o$C(b) 600$^o$C**

Examination of Image exhibits that flecks are polycrystalline, cluttered in style and
2D nano thin film in formation with flecks like structure.

## CONCLUSIONS

The nano-sized stuff of Zinc Oxide doped $MnO_2$ having various calcinations temperature of the Zinc Oxide has been assembled by Micro-wave treated co-precipitation advance synthesis protocols. The conclusions of HRTEM assist the XRD sequels grain size approximate to 26 to 38 nm. The Perusal of HRTEM images exhibit that the size of all ignited Zinc Oxide doped $MnO_2$ nano-networked specimen lies in a confine of 26 nm to 38 nm and 2-D nanosheets were in formation. The FTIR Spectra of the ignited nano networked stuff of Zinc Oxide doped $MnO_2$ containing calcinations temperature was exhibiting the Alps(peaks) at positions 516 $cm^{-1}$ and 837 $cm^{-1}$, Mn-O-Mn and Zn-O-Zn vibrations were identified. The M-H curve study of calcined samples concluded that nature of all calcined samples were paramagnetic in nature and magnetic susceptibility shows inverse behaviour with rise of temperature and follow Curie-Weiss Law of magnetism.

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