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ENVIRONMENTAL FRIENDLY WET CHEMICAL SYNTHESIS AND CHARACTERIZATION OF TRANSITION METAL DOPED PARTICLE

Arti Gupta* and Anjali Pandey

Department of Chemistry, C.M.P. College, University of Allahabad, Allahabad - 211002, Uttar Pradesh, India.

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Correspondence to Author:
Dr. Arti Gupta

Associate Professor,
Department of Chemistry,
C.M.P. College, University of Allahabad, Allahabad - 211002,
Uttar Pradesh, India.

E-mail: dr.artig@yahoo.in

ABSTRACT: Transition metal doped zinc oxide particles were synthesized in aqueous phase adopting a low cost, easy and environmental friendly route. The precursors used in the preparation of Co doped ZnO ($Zn_{0.95}Co_{0.05}O$) and ZnO ($Zn_{0.90}Co_{0.1}O$) by wet process are zinc acetate dehydrates cobalt chloride, sodium hydroxide, di-ethylene glycol and double distilled water. The doped ZnO particles were characterized by using scanning electron microscope (SEM), energy dispersive X-ray (EDX), Fourier transforms infrared spectroscopy (FTIR). The study constitutes the basis for developing versatile applications of transition metal doped ZnO microstructures.

INTRODUCTION: Transition metal doped micro structure is an effective method to adjust the energy level surface states of ZnO by the changes in doping concentrations of doped materials and hence in its physical and optical properties [1]. Pure ZnO is an eco-friendly material moreover non toxic for human bodies and many of its compounds may be useful in bio-medical applications. ZnO clusters are large band gap semiconductor and they can be produced by various ways [2]. ZnO, a direct wide band gap (3.4 eV at Room temperature) compound n-type semiconductor, has a stable wurtzite structure with lattice spacing a = 0.325 nm and c = 0.521 nm and composed of a number of alternating planes with tetrahedrally-coordinated O^{2-} and Zn^{2+} ions, stacked alternately along the c-axis.

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It has a unique position among semiconducting oxides due to piezoelectricity and transparent conducting properties. Its semiconducting property has been favoured by good transparency, high electron mobility, wide band gap, strong room temperature, ultraviolet light emitters, chemical sensors, spintronics and luminescence [3-12]. All these predominant properties make ZnO a great potential in the field of nanotechnology. Research on ZnO has continued for many decades following a roller-coaster pattern.

Lattice parameters of ZnO, optical properties and processes in ZnO as well as its refractive index, vibrational properties by techniques such as Raman scattering were extensively investigated. Nanostructures ZnO have many potential applications in photocatalysis [13, 14], solar cell [15, 16], gas sensors [17, 18], fuel cells [19], photovoltaics [20], antibacterial action [21]. For practical applications, the proper dopants may be introduced into ZnO and various properties of ZnO can be influenced by the chemical doping or formed intrinsic lattice defects. ZnO doped with several 3d transition metal ions such as V, Cr, Fe and Ni have been reported for their ferromagnetic properties. The doped ZnO would be suitable for number of devices such as spin field emission transistors (FETs) and light emitting diodes (LEDs) with circularly polarized light emission [22]. Co-doping in ZnO progressively decreased the photocatalytic activity while the Mn-doping initially increased activity. Wet chemical methods are attractive for their low cost, less hazardous effect; capable of easy scaling up [23] growths occurs at a relatively low temperature, compatible with flexible organic substrates, no need for the use of metal catalysts and can be integrated with well developed silicon technologies [24].

Various types of micro structured materials are synthesized by using different physical methods such as simple vapour transport and condensation process, sol-gel method, solid state reaction method, radio-frequency magnetron sputtering technique, facile low temperature synthesis, chemical co-precipitation method [17-20] etc. In the aqueous phase or wet process synthesis of cobalt doped zinc oxide, the $Zn_{1-x}Co_xO$; x=0.05-0.1 particles were successfully synthesized.

Experimental:

MATERIALS AND METHODS:

Synthesis of Cobalt Doped Zinc Oxide ($Zn_{1-x}Co_xO$; x=0.05.0.1) Particles: $Zn(Ac)_2.2H_2O$, $CoCl_2$ were used as precursors for preparation of doped particles in wet chemical method. Di-ethylene glycol and sodium hydroxide were used for the homogeneity and pH adjustment of the solution and helps to make a stoichiometric solution to get zinc oxide particles. The ZnO powder obtained from this method was calcined at temperature 100 °C. For preparing 5% Co doped ZnO ($Zn_{0.95}Co_{0.05}O$) and 10% Co doped ZnO ($Zn_{0.9o}Co_{0.1}O$), zinc acetate and cobalt chloride were taken in a beaker containing distilled water according to calculated stoichiometric ratio.

Diethylene glycol was added to above solution with continuous stirring. NaOH solution was added dropwise and stirred till the pH was maintained to 10-12. The solution was divided into three parts. One part was heated at 100 °C for thirty minutes, second part for sixty minutes and the third part for ninety minutes. It was filtered, washed by distilled water and then by ethanol. The precipitate was dried in the oven and weighed.

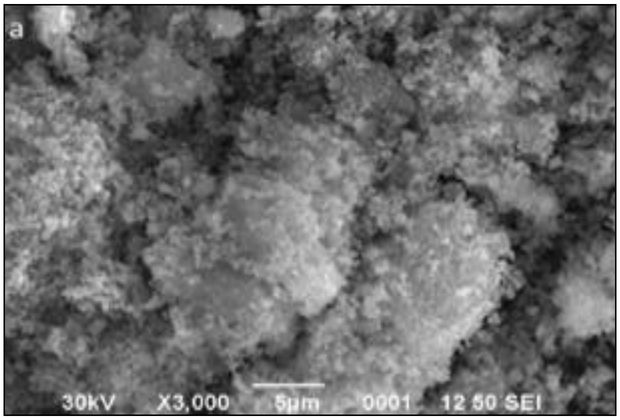
$$Zn^{2+} + 2OH^- \longleftrightarrow Zn(OH)_2 \quad \text{----------1}$$
$$Zn(OH)_2 + 2OH^- \longleftrightarrow [Zn(OH)_4]^{2-} \quad \text{----------2}$$
$$[Zn(OH)_4]^{2-} \longleftrightarrow ZnO_2^{2-} + 2H_2O \quad \text{----------3}$$
$$ZnO_2^{2-} + H_2O \longleftrightarrow ZnO + 2OH^- \quad \text{----------4}$$
$$ZnO + OH^- \longleftrightarrow ZnOOH^- \quad \text{----------5}$$

Thus, in the aqueous phase or wet process synthesis of cobalt doped zinc oxide, the $Zn_{1-x}Co_xO$; x=0.05.0.1 doped particles were successfully synthesized.

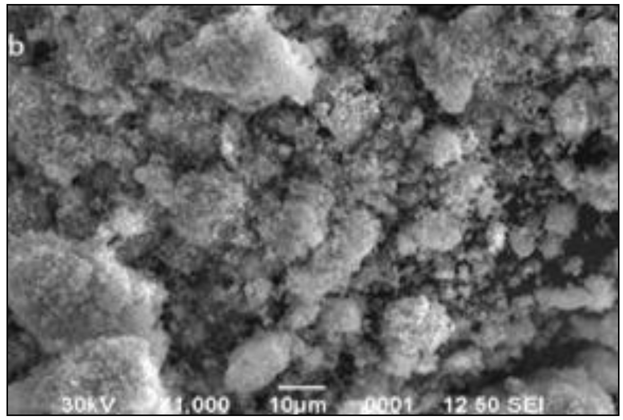
Characterization: In order to investigate various properties of the prepared sample, numbers of characterizations techniques have been performed. The results show the different optical and structural properties of the prepared sample. Chemical composition and morphology of the samples were carried out using a scanning electron microscope, SEM (JSM-6490 LV) equipped with Energy Dispersive X-ray (EDX) thermo Electron Corporation. The mean particle size and corresponding standard deviation of the ZnO particles were determined by image analyses of SEM micrographs. FTIR (IR Spectrometer, Hicolet™–6700 of thermo-scienctific USA) was used to study absorbance properties.

RESULTS AND DISCUSSION: Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDX) and Fourier Transforms Infrared Spectroscopy (FTIR) methods were employed to characterize the cobalt doped zinc oxide, the $Zn_{1-x}Co_xO$; x=0.05.0.1 particles. SEM micrographs (**Fig. 1**) revealed spherical sub micro-particles; however there is wide size distribution from 0.2µm to 1 µm. Further, spherical particles tending to attain one dimentional(1-D) structures (**Fig. 1: a, b, c, d, e, f, g, h and k**).

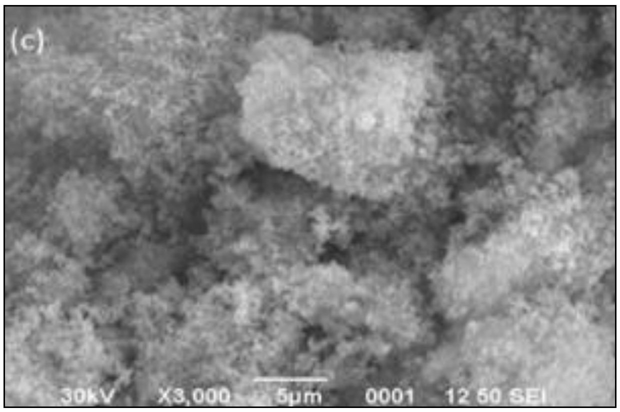
Morphology and elemental analysis were carried out by Energy Dispersive Spectra (EDX). EDX (**Fig. 2)** data shows composition of Co, Zn are present in sample and other impurities such as Mg, Ca, Al, Si ,C and O present in atmosphere. Zn: Co: O ratio has been calculated to be 0.95:0.05:1 and 0.90:0.0.1:1 for 5% Co-doped ZnO and 10% Co-doped ZnO respectively. Other peaks correspond to Al, Ca, Mg, Si, C and O is seen due to some impurities.



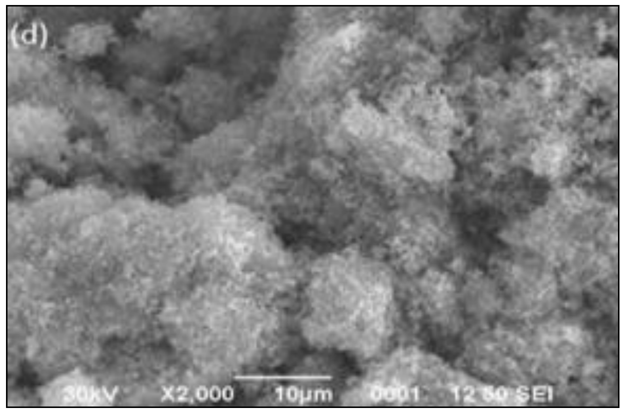
5% Co DOPED ZnO HEATED AT 100 °C TO 30 MIN



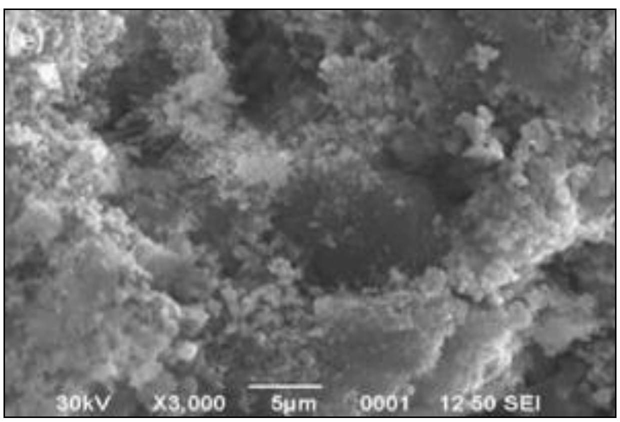
5% Co DOPED ZnO HEATED AT 100 °C TO 30 MIN



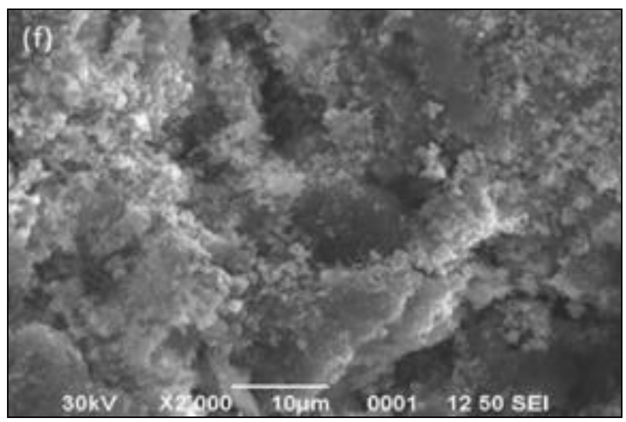
5% Co DOPED ZnO HEATED AT 100 °C TO 60 MIN



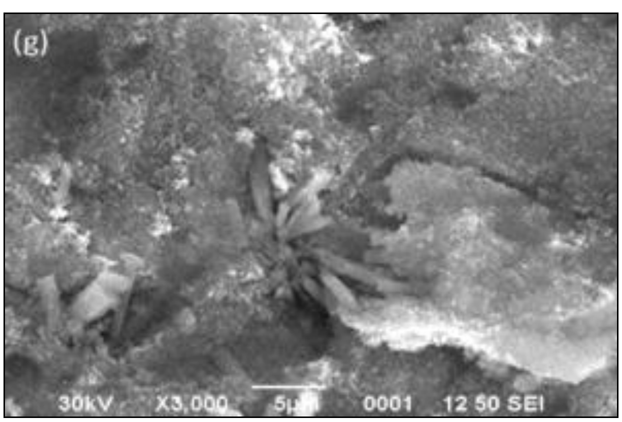
5% Co DOPED ZnO HEATED AT 100 °C TO 60 MIN



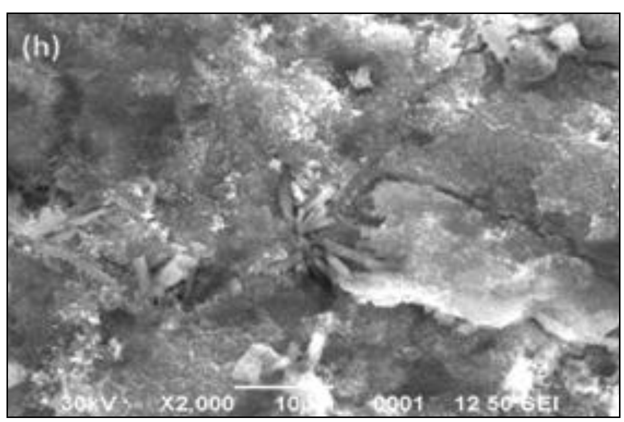
5% Co DOPED ZnO HEATED AT 100 °C TO 90 MIN



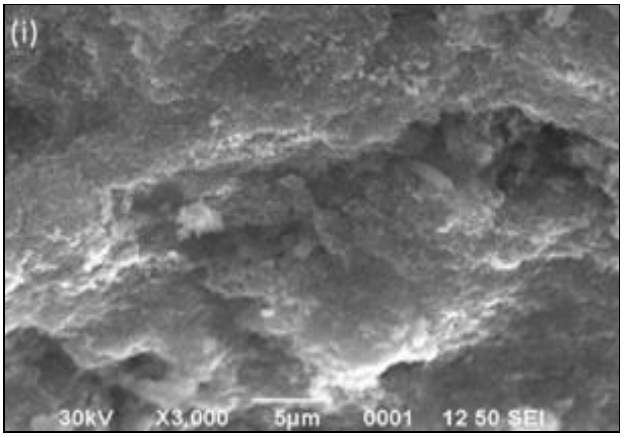
5% Co DOPED ZnO HEATED AT 100 °C TO 90 MIN



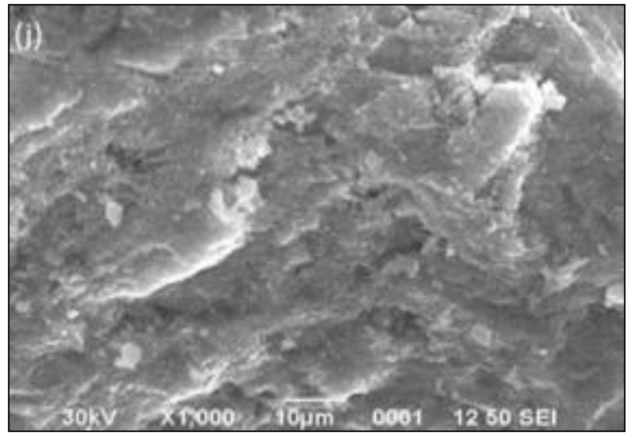
10% Co DOPED ZnO HEATED AT 100 °C TO 30 MIN



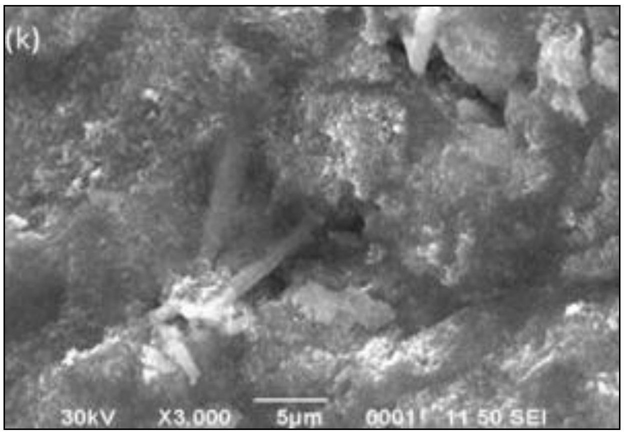
10% Co DOPED ZnO HEATED AT 100 °C TO 30 MIN



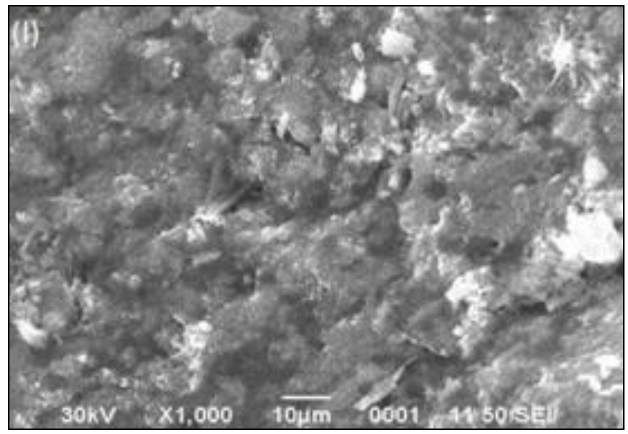
10% Co DOPED ZnO HEATED AT 100 °C TO 60 MIN



10% Co DOPED ZnO HEATED AT 100 °C TO 60 MIN

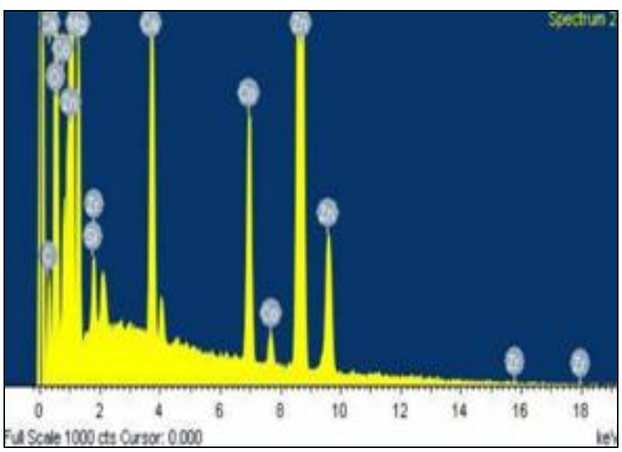


10**% Co DOPED ZnO HEATED AT 100 °C TO 90 MIN**

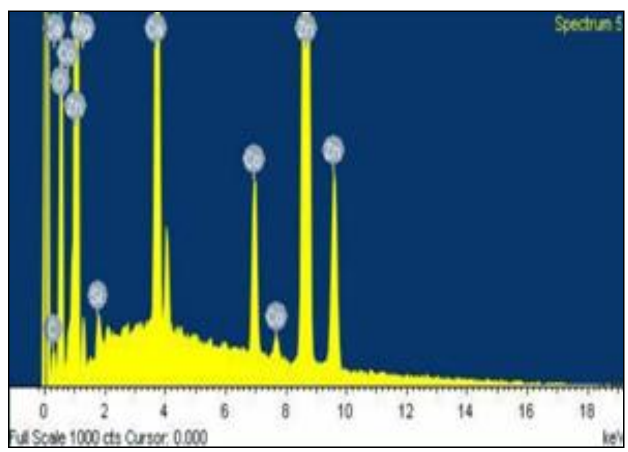


10% Co DOPED ZnO HEATED AT 100 °C TO 90 MIN

FIG. 1: SEM MICROGRAPH FOR 5%, 10% Co-DOPED ZnO HEATED AT TEMPERATURE 100 °C TO DIFFERENT TIME 30, 60 AND 90 MINUTES AT DIFFERENT RESOLUTION OF SEM



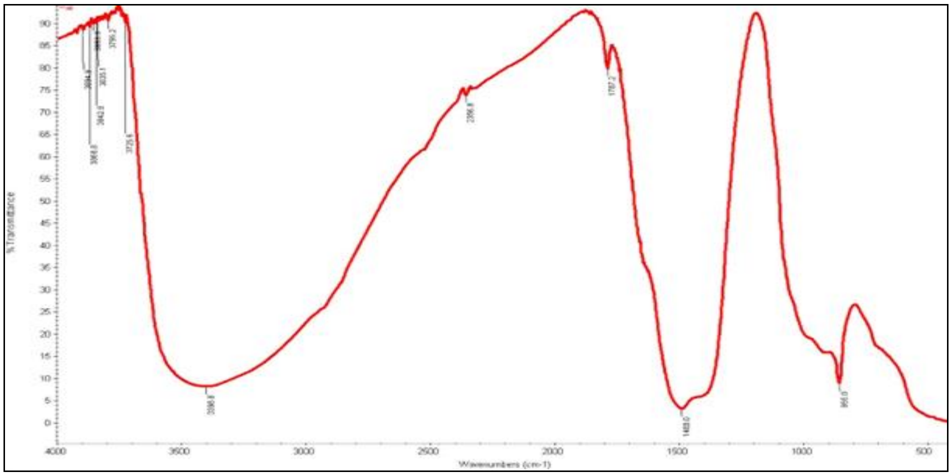
5% Co DOPED ZnO HEATED AT 100 °C TO 30 MIN



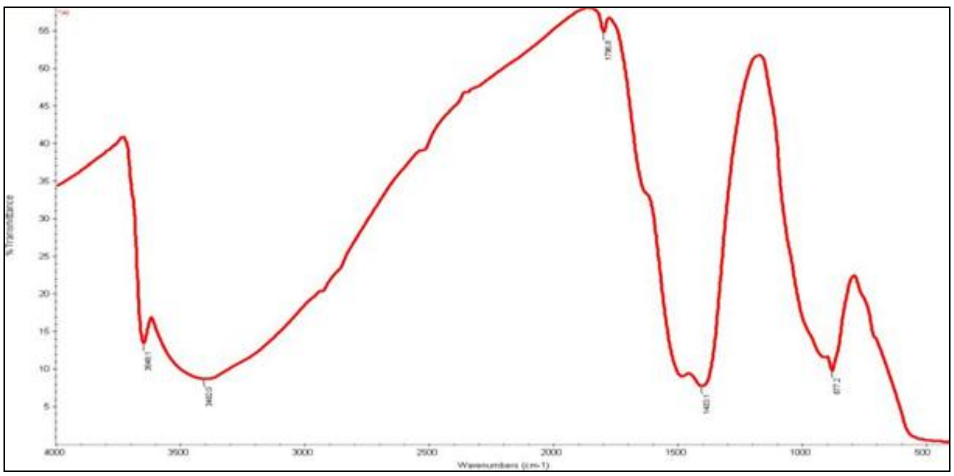
10% Co DOPED ZnO HEATED AT 100 °C TO 30 MIN

FIG. 2: EDX IMAGE AND SPECTRA OF Co-DOPED ZnO PARTICAL WITH DIFFERENT CONCENTRATION 5%, 10% OF Co IN ZnO AT 100 °C

FTIR pattern (**Fig. 3**) showed the broad and intense peak at 3398 cm^{-1} corresponds to O-H due to presence of hydrated H_2O molecules. Apart from this duplet peaks at 1486 cm^{-1}, 1394 cm^{-1} due to CH_2 bending and CH_2 wagging of diethylene glycol molecules. The other peak at 855 is due to C-C stretching of diethylene glycol. All the peak corresponds to diethylene glycol shows some downward shifts as compared to pure diethylene glycol indicating adsorption of diethylene glycol at doped ZnO particles surface.



FTIR PROFILE 5% COBALT-DOPED ZnO AT 100 °C



FTIR PROFILE 10% COBALT-DOPED ZnO AT 100 °C

FIG. 3: FTIR SPECTRA OF Co-DOPED ZnO PARTICLES

Nanocrystals of 5% and 10% Co doped ZnO particles were successfully prepared by wet chemical aqueous phase synthetic method and characterised by SEM, EDX and FTIR. The effects of dopant contents and synthesis process on the structural and FTIR pattern of the products were discussed. Further studies on this environmentally benign system may provide many interesting aspect in the field of material science.

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CONFLICTS OF INTEREST: Nil

REFERENCES:

1. Nomura K, Ohta H, Ueda K, Kamiya T, Hirano M and Hosono H: Thin-film transistor fabricated in single-crystalline transparent oxide semiconductor. Science 2003; 300: 1269.
2. Nakada T, Hirabayashi Y, Tokado T, Ohmori D and Mise T: Novel Device Structure for Cu(In, Ga)Se2 Thin Film Solar Cells Using Transparent Conducting Oxide Back and Front Contacts. Solar Energy Sol. Energy 2004; 77: 739.
3. Lee SY, Shim ES, Kang HS, Pang SS and Kang JS: Thin Solid Films 2005; 437: 31.
4. Könenkamp R, Word RC and Schlegel C: Vertical nanowire light emitting diode Appl. Phys. Lett 2004; 85: 6004.
5. Mckinstry ST and P Muralt: J Electroceram. 2004; 12: 7.
6. Wang ZL, Kong XY, Ding Y, Gao P, Hughes WL, Yang R and Zhang Y: Semiconducting and piezoelectric oxide nanostructures induced by polar surfaces. Adv. Funct. Mater 2004; 14: 943.
7. Wagh MS, Patil LA, Seth T and Amalnerkar DP: Surface cupricated SnO 2–ZnO thick films as a H_2 S gas sensor. Mater. Chem. Phys 2004; 84: 228.
8. Ushio Y, Miyayama M and Yanagida H: Effects of Interface States on Gas Sensing Properties of a CuO / ZnO Thin Film Heterojunction Sensor Actuat. B 1994; 17: 221.
9. Harima H: J. Phys. Condens. Matter 2004; 16: S5653.
10. Pearton SJ, Heo WH, Ivill M, Norton DP and Steiner T: Dilute magnetic semiconducting oxides Semicond. Sci. Technol 2004; 19: R59.
11. Han J, Senos AMR and Mantas PQ: Deep Donors in Polycrystalline Mn-doped ZnO Mater Chem. Phys 2002; 117-120.
12. Jakani M, Campet G, Claverie J, Fichou D, Pouliquen J, and Kossanyi J: Photoelectrochemical properties of zinc

oxide doped with 3d elements. Journal of Solid State Chemistry J. Solid State Chem 1985; 269-277.
13. Xia SS, Zha L, Leng XN, Lang XY and Lian JS: Synthesis of Amorphous TiO_2 Modified ZnO Nanorod Film with Enhanced Photocatalytic Properties. Applied Surface Science 2014; 299: 97-104.
14. Kundu SA: Facile Route for the Formation of Shape Selective ZnO Nanoarchitectures with Superior Photocatalytic Activity. Colloids and Surfaces A: Physiochemical and Engineering Aspects 2014; 446: 199-212.
15. Huang J, Yin ZG and Zheng QD: Applications of ZnO in Organic and Hybrid Solar Cells. Energy and Environmental Science 2011; 4: 3861-3877.
16. Xu F and Sun LT: Solution-Derived ZnO Nanostructures for Photoanodes of Dye-Sensitized Solar Cells. Energy and Environmental Science 2011; 4: 818-841.
17. Wang PP, Qi Q, Xuan RF, Zha J, Zhou LJ and Li GD: A Facile Method for Enhancing the Sensing Performance of Zinc Oxide Nanofibers Gas Sensors. RSC Advances 2013; 3: 19853-19856.
18. Alenezi MR, Henley SJ, Emerson NG and Silva SRP: From 1D and 2D Nanostructures to 3D Hierarchical Structures with Enhanced Gas Sensing Properties. Nanoscale 2014; 6: 235-247.
19. Qin Y, Wang XD and Wang ZL: Microfiber-Nanowire Hybrid Structure for Energy Scavenging. Nature 2008; 451: 809-813.
20. Song HS, Zhang WJ, Cheng C, Tang YB, Luo LB, Chen X, Luan CY, Meng XM, Zapien JA, Wang N, Lee CS, Bello I and Lee ST: Controllable Fabrication of Three-Dimensional Radial ZnO Nanowire / Silicon Microrod Hybrid Architectures. Crystal Growth and Design 2011; 11: 147-153.
21. Coey JMD, Venkatesan M and Fitzgerald CB: Donor impurity band exchange in dilute ferromagnetic oxides. Nat. Mater 2005; 173.
22. Sudakar C, Thakur JS, Lawes G, Naik R and Naik VM: Ferromagnetism induced by planar nanoscale CuO inclusions in Cu-doped ZnO thin films Phys. Rev 2007; B75: 054423.
23. Zeng HB, Xu XJ, Bando Y, Gautam UK, Zhai TY, Fang XS, Liu BD, Golberg and Shah D: Template deformation-tailored ZnO nanorod / nanowire arrays: full growth control and optimization of field-emission. Adv. Funct. Mater 2009; 19: 3165.
24. He M, Tian YF, Springer D, Putra IA, Xing GZ, Chia EEM, Cheong SA and Wu T: Polaronic transport and magnetism in Ag-doped ZnO Appl. Phys. Lett 2011; 99: 222511-1.

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