

Synthesis and characterization of CeO-NiO nanocomposites

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ABSTRACT

A CeO-NiO nanocomposites have been synthesized by a Sol-Gel method based on polymeric network of polyvinyl alcohol (PVA). In this method mixture solvent of 50:50 ethanol- water was used to dissolve cerium nitrate, nickel nitrate and PVA. The mixture was heated to 80°C to form homogeneous gel solution. The obtained gel was slowly heated to evaporate the solvent to form a hard homogeneous gel. The hard gel was calcinated at a temperature of 600°C for 4 hours and 8 hours and converted into nanocomposites. The prepared nanocomposites have been characterized using X-Ray Diffraction, SEM, FTIR, UV-VIS. The size of a nanocomposites heated at 600°C for 4 hours and 8 hours using scherrer formula comes out to be 17 nm and 18.3nm. TEM images of nano composite confirms that the nanoparticles formed are in nanosize. At 4h spectra, absorption first decreases in ultraviolet region and there is absorption peak at 240 nm wavelength. Then absorption peak increase a little in UV region and there is second absorption peak at about 310 nm. It increases further little in UV region and then decreases in the visible region up to 800 nm. At 8 h spectra, absorption first decreases fast and there is peak at 220 nm and then it rises exponentially up to 400 nm in ultraviolet region. Absorption curve then remain constant up to 580 nm and then decreases fast in the visible region. Infrared spectroscopy is used to determine presence of certain functional group. The nanocomposite result was confirmed with TEM analysis. FTIR spectra shows new peaks at 3838 cm⁻¹, 3740 cm⁻¹, 1624 cm⁻¹, and 412 cm⁻¹ when calcinated for 8 h.

Key Words: Nanocomposites, Synthesis, XRD, FTIR, UV-VIS, SEM

INTRODUCTION

A rare earth oxide such as CeO₂ has been applied widely in many fields. Cerium oxide is used as catalysis field and in chemical and mechanical polishing. Cerium oxide is also well known for its optical properties and it can filter ultraviolet (UV) rays. Cerium oxide is a semiconductor with a band gap of around 3.0 -3.2 eV.[1-3] NiO has a large exciton binding energy and having a large band gap ranging from 3.6 to 4.0 eV. This is a p-type semiconductor and can be used in optical, catalytic and electronic devices like solar cells, gas sensors etc.

NiO nanoparticles have many unique optical, chemical, electrical and magnetic properties. Nanometer-sized NiO is expected to possess many better properties than those of bulk NiO. NiO nanoparticles are much more effective catalysts than commercial NiO powder for catalytic reduction of carbon dioxide to methanol. NiO nanoparticles have applications in the field of ceramic materials, electronic components, sensors, magnetic data storage materials and catalysts. NiO has many applications in solar thermal absorber, catalyst for oxygen evolution, photo electrolysis and electro chromic device. [4-10]

The study of composite material ie mixture consisting of at least two phases of different chemical composition. It has been of great interest from both fundamental and practical point of view. The physical properties of such

materials can be combined to produce material of desired response. Optical or magnetic characteristics can change upon the decrease of particle sizes to very small dimensions, which are, in general, of major interest in the area of nanocomposite materials. Composites have excellent properties such as high hardness, high melting point, low density, low coefficient of thermal expansion, high thermal conductivity, good chemical stability and improved mechanical properties such as higher specific strength, better wear resistance and specific modulus and have good potential for various industrial fields.[11-14] The synthesized nanoparticles were characterized by XRD, TEM and UV-VIS, FTIR spectrometer.

MATERIALS AND METHODS

In this work mixed ethanol-water solvent (50:50) was used to dissolve 2 gm cerium nitrate, 2 gm nickel nitrate and 8 gm PVA. The mixture was heated at 80°C to form a homogeneous gel solution. The obtained sol was slowly heated to evaporate the solvent and it form a hard homogeneous gel. The pyrolysis of the final gel was performed at a temperature of 600°C for 4 hours and 8 hours. During the pyrolysis process the PVA polymeric network through the outer surface, cerium and copper nitrate salt simultaneously calcinated and converted into CeO-NiO nanocomposites. The obtained sample were crushed to prepare a fine powder.

RESULTS AND DISCUSSION

XRD Studies:-

The XRD pattern of CeO-NiO nanocomposites shown in Fig 1 and 2 which were calcinated at 600°C for 4 hours and 8 hours were obtained using panalytical's Xpert-pro powder diffractometer employing Cu -K α radiations in the 2 θ range 10^o-90^o.The particle size of as prepared samples were found using Scherrer formula using Scherrer formula

$$d = 0.9\lambda / \beta \cos\theta$$

Where d= average particle size, β is full width at half maxima(FWHM), θ is the Bragg angle, λ is the wavelength of Cu K in radians [15-20]

It is seen from XRD that particle size increases when we increase the time of calcinations. The particle size when calcinated at 600°C for 4 h is 16.3 nm whereas when calcinated at 600°C for 8 h comes out to be 17 nm

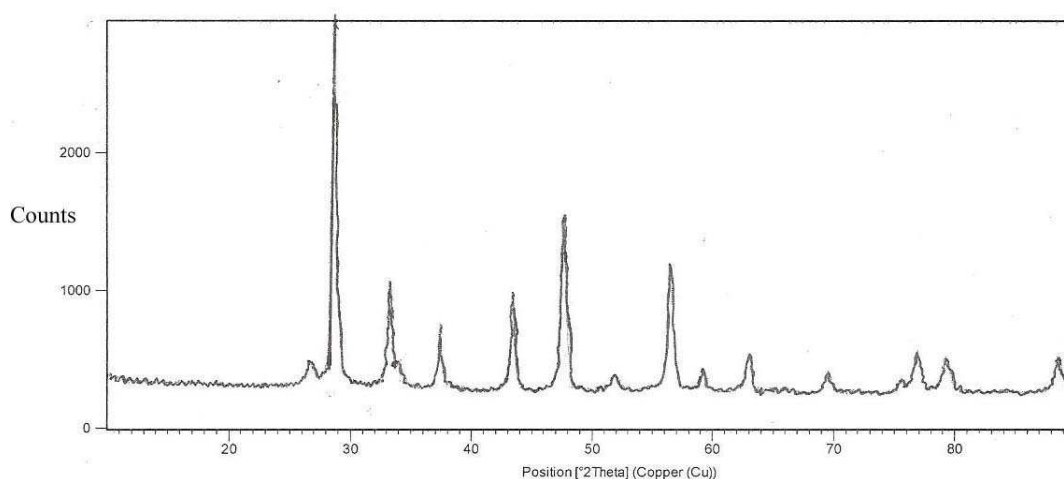


Fig 1. XRD Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 4 h

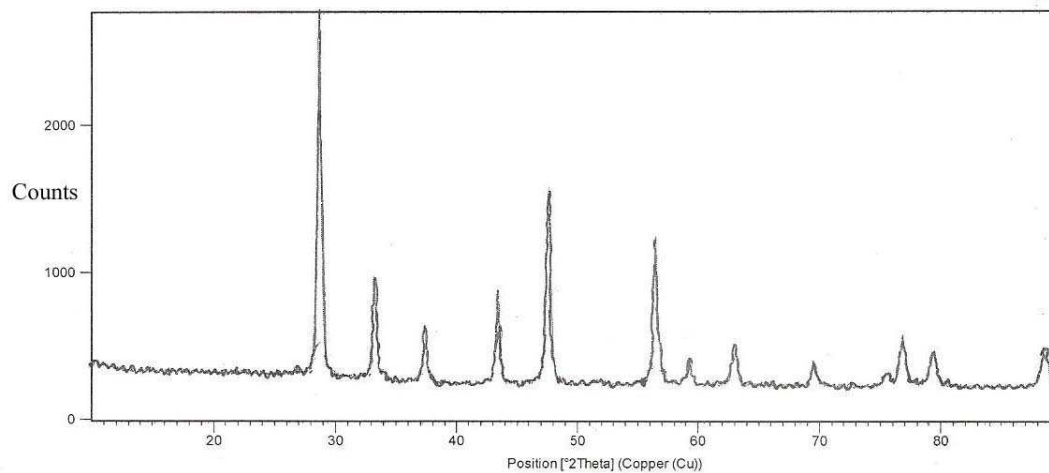


Fig 2. XRD Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 8 h

TEM:-

TEM analysis provide information on the size and morphology of CeO-NiO nanocomposites and their state of agglomeration. TEM images of CeO-NiO Nanocomposites annealed for 4h and 8 h at 600°C are shown in Fig 3 and 4. The image indicate that the nanoparticles of CeO-NiO which were calcinated at 600°C for 4 h have a hexagonal shape and are in the form of spherical shape and are well dispersed with smooth surface of uniform size. Nanoparticles of CeO-NiO which were calcinated at 600°C for 8 h consisted of separated nanoparticles of various sizes from the afore-mentioned range as well as it from clusters of small nanoparticles. The TEM image shows a homogenous particle distribution [21-23]

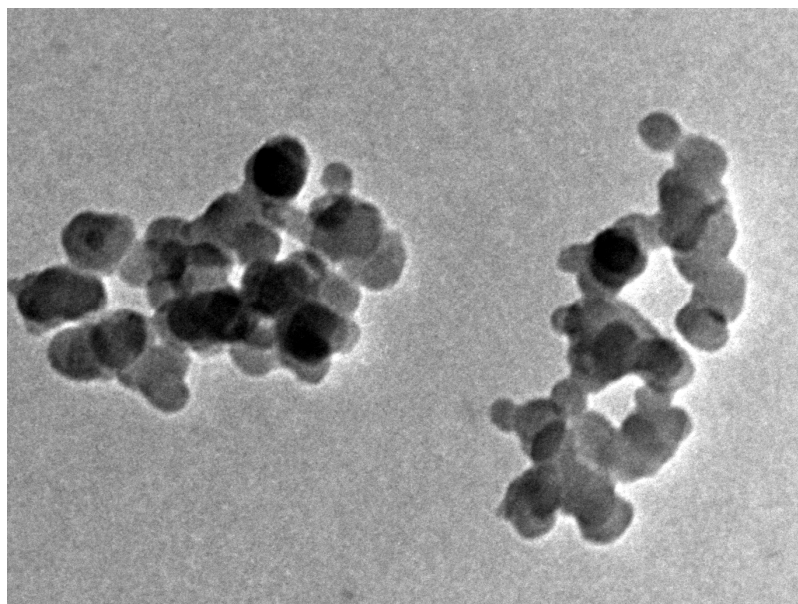


Fig 3. TEM Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 4 h

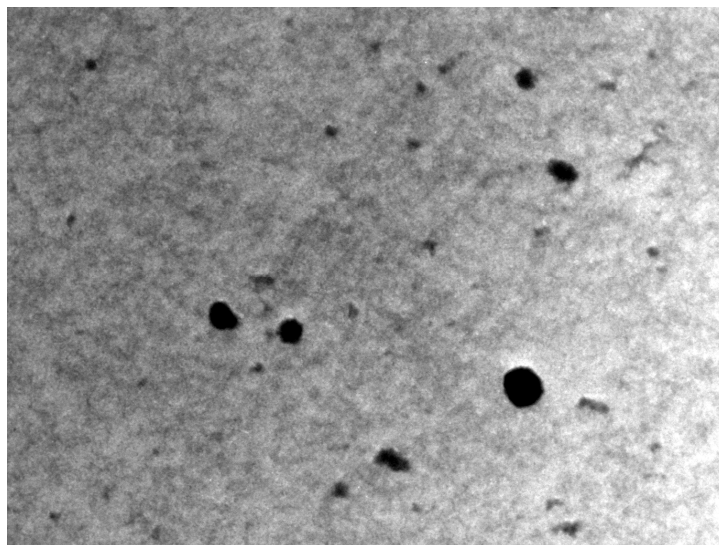


Fig 4. TEM Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 8 h

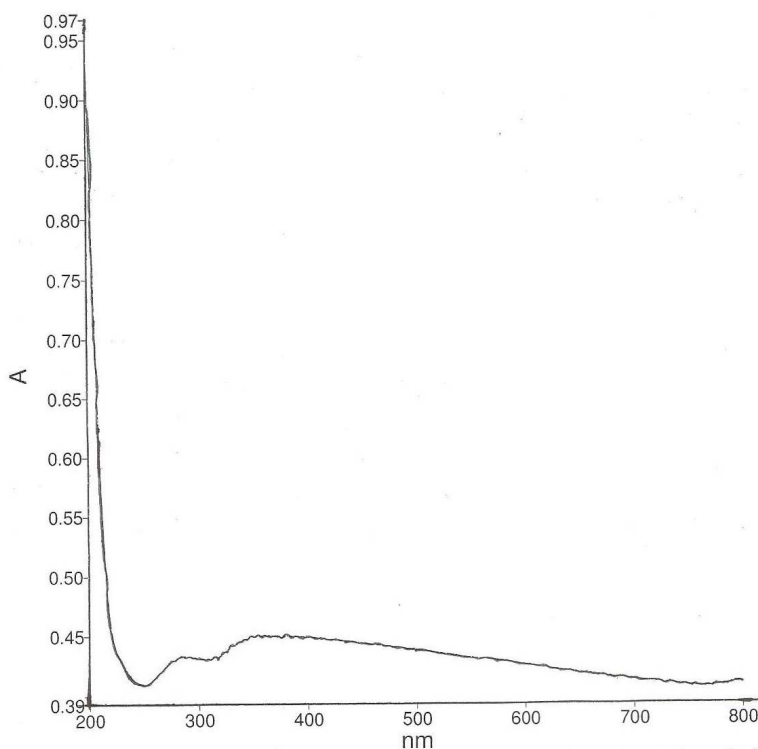


Fig 5 UV-VIS Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 4 h

UV-VIS Spectral Studies:-

The UV-VIS study of nanocomposites CeO-NiO were done using Hitachi 330 spectrophotometer in the wavelength range 200 to 800 nm. The spectra of nanocomposites calcinated for 4h and 8 h at 600°C are shown in Fig 5 and 6.

At 4h spectra, absorption first decreases in ultraviolet region and there is absorption peak at 240 nm wavelength . Then absorption peak increase a little in UV region and there is second absorption peak at about 310 nm. It increases

further little in UV region and then decreases in the visible region up to 800 nm. At 8 h spectra, absorption first decreases fast and there is peak at 220 nm and then it rises exponentially up to 400 nm in ultraviolet region. Absorption curve then remain constant up to 580 nm and then decreases fast in the visible region. [24-28]

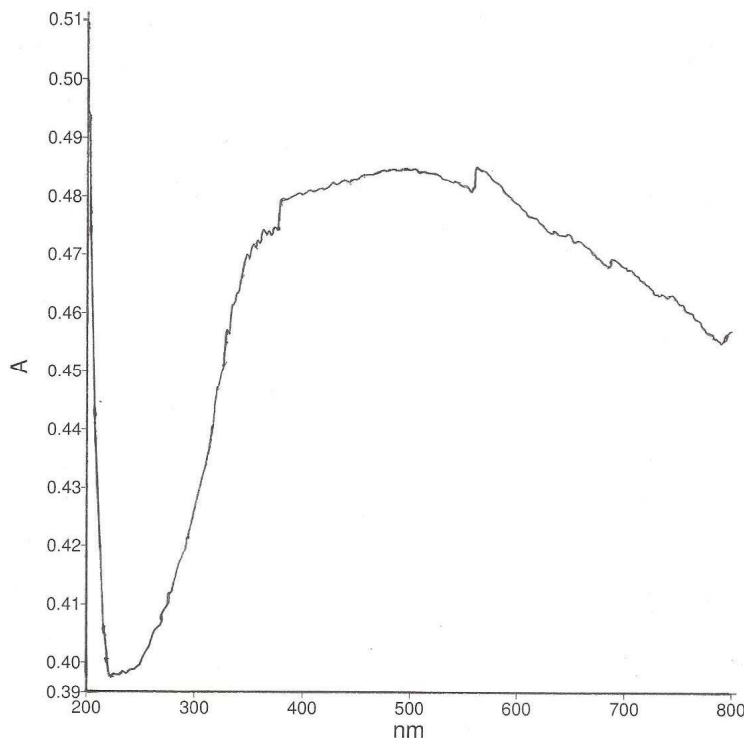


Fig 6 UV-VIS Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 8 h

FTIR Spectra Analysis:-

The infrared spectroscopic (IR) study of the nanocomposites were done using Perkin Elmer –Spectrum RX FTIR Spectrophotometer in the wave number range 400-4000 cm^{-1} . The FTIR spectra of CeO-NiO nanocomposites are shown in Fig 7 and 8. Infrared spectroscopy is used to determine presence of certain functional group. FTIR spectra calcinated for 4 h at 600°C shows peaks at 3420 cm^{-1} , 2915 cm^{-1} , 2849 cm^{-1} , 1609 cm^{-1} , 1021 cm^{-1} and 532 cm^{-1} . A broad band at 3420 cm^{-1} corresponds to stretching mode of OH group which is contributed by water contents. The peak at around 2900 cm^{-1} is due to C-H band. Band around 1600 cm^{-1} may be due to deformation vibration of H₂O molecule. Bands around 1100 cm^{-1} may be due to single C-O bond stretching mode. Band spectral region around 532 cm^{-1} may be assigned to Ni-O stretching vibrational mode.

FTIR spectra calcinated for 8 h at 600°C shows peaks at 3838 cm^{-1} , 3740 cm^{-1} , 3399 cm^{-1} , 1624 cm^{-1} , 1021 cm^{-1} , 726 cm^{-1} , 510 cm^{-1} , 412 cm^{-1} . Bands at 3399 cm^{-1} , 3838 cm^{-1} , 3740 cm^{-1} corresponds to stretching mode of OH group which is contributed by water contents. Band near 1624 cm^{-1} is assigned to H-O-H bending vibrational mode due to absorption of water in air. Bands around 1100 cm^{-1} may be due to single C-O bond stretching mode. Band spectral region around 510 cm^{-1} may be assigned to Ni-O stretching vibrational mode. [29-32]

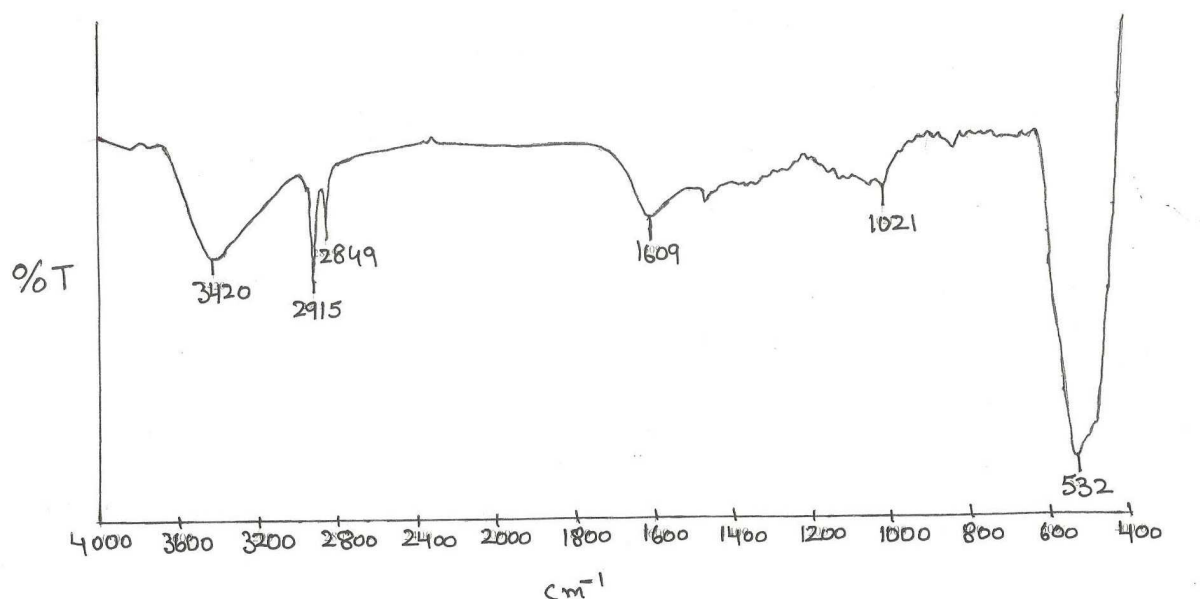


Fig 7 FTIR Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 4 h

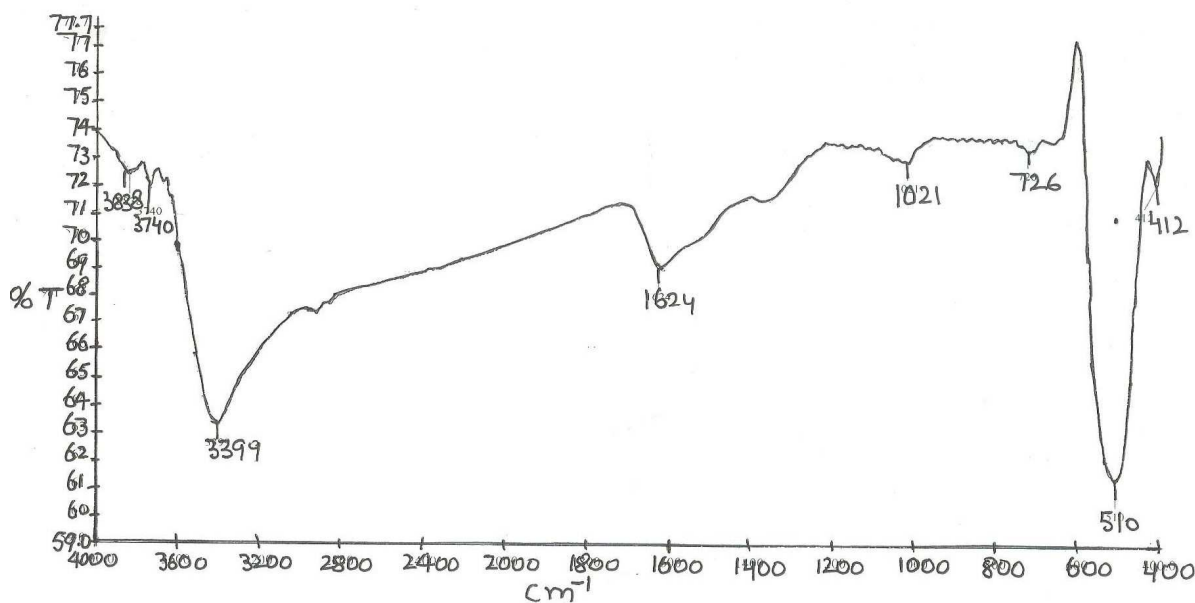


Fig 8 FTIR Spectra of CeO-NiO Nanocomposite Calcinated at 600°C for 8 h

CONCLUSION

CeO-NiO nanocomposites have been prepared successfully by sol-gel method. The size of the nanocomposites so formed using XRD Scherrer formulae comes out to be 16.3 nm and 17 nm when calcinated at 600°C for 4h and 8h. The UV-VIS shows increasing absorption at the edge of UV-VIS and UV region and then decreasing in visible region more when calcinated at 8h. The nanocomposite result was confirmed with TEM analysis. FTIR spectra shows new peaks at 3838 cm^{-1} , 3740 cm^{-1} , 1624 cm^{-1} , and 412 cm^{-1} when calcinated for 8 h.

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REFERENCES

- [1] Bhaskar Chander Mohanty et al., *Material Research Bulletin*, **2011**, 46, 875.
- [2] L. Truffault, M.-T. Ta, T. Devers, K. Konstantinov, V. Harel, C. Simmonard, C. Andreazza, I. P. Nevirkovets, A. Pineau, O. Veron, and J. P. Blondeau, *Mater. Res. Bull.*, **2010**,45, 527.
- [3] L. Zhou, W. Wang, S. Liu, L. Zhang, H. Xu, and W. Zhu, *J. Mol. Catal. A Chem.*, **2006**, 252, 120.
- [4] Li Y, Cai M, Rogers J, Xu Y, Shen W, *Mater. Lett.*, **2006**, 60, 750.
- [5] Thakur M, Patra M, Majumdar S, Giri S, *J. Alloys Compd.*, **2009**,480, 193.
- [6] Johnston-Peck A.C, Wang J, Tracy J. B, *ACS Nano*, **2009**, 3, 1077.
- [7] Sun X. C, Dong X. L, *Mater. Res. Bull.*, **2002**, 37, 991.
- [8] Roy A, Srinivas V, Ram S, De Toro J.A, Mizutani U, *Phys. Rev. B* **2002**,71, 184443
- [9] H. He, R. H. Heist, B. L. McIntyre and T. N. Blanton, *NanoStructured Materials*, **1997**, 8, 879.
- [10] Z. J. Suh, H. D. Jang, H. K. Chang, D. W. Hwang and H. C. Kim, *Materials Research Bulletin*, **2005**,40, 2100.
- [11] Ashwani Sharma, Pallavi, Sanjay, *Nano Vision*, **2011**,1, 115
- [12] Sanjay Kumar, Ashwani Sharma, M. Singh, Pooja Dhimen, R. K. Kotnala, *Nano Vision* **2011**,1 101.
- [13] Ashwani Sharma, Pallavi, Sanjay kumar, *Nanoscience and nanotechnology* **2012**,12, 82.
- [14] Ashwani Sharma, Pallavi, Sanjay Kumar, *Research journal of pharmaceutical biological and chemical science RJPBCS* **2012**,3 1340.
- [15] Hofmeister H, Tan G.L and Dubeil M, *J. Mater.*, **2005**, **20**, 55.
- [16] Hammond C The Basic of Crystallography and Differaction; Oxford University Press; New York, NY, 1997.
- [17] Zang H. and Lacefield W. R. , *Biomaterials*, **2000**, 21, 23
- [18] Okereke, N. A and Ekpunobi, A. J. *Pelagia Research Library -Advances in Applied Science Research*, **2012**, 3 (3), 1244-1249.
- [19] Patil, T.K. and Talele, M. I, *Pelagia Research Library-Advances in Applied Science Research*, **2012**, 3 (3), 1702-1708.
- [20] S. Srikantha, N. Suriyanarayananb, S. Prabahara, V. Balasubramaniana, D. Kathirvelc., *Pelagia Research Library-Advances in Applied Science Research*, **2011**, 2 (1): 95-104
- [21] G.D. Guthrie, D.R. Veblen, *Clays Clay Miner.*, **1989**,37 1.
- [22] Goldstein A. N, Echer C. M, Alivisatos A. P, *Science*, **1992**, 256, 5062.
- [23] Song, J.Y. and B. S. Kim, *Korean. J. chem. Eng.*, **2008**, **25**(4). 808
- [24] Suresh S., and Anand K., *Advances in Applied Science Research*, **2012**, 3 (2):815-820
- [25] Jeroh M. D. and Okoli D. N., *Advances in Applied Science Research*, **2012**, 3 (2):793-800
- [26] J. I. Pancove. *Optical Processes in Semiconductors* Englewood Chiffs. NJ Prentice Hall (1971).
- [27] Gosain D. P, Shimizu T, Suzuki M, Bando T, and Okano S, *J. Mater Sci.* **2001**,26 ,3271.
- [28] E. Borrodo, F., Prieto, J, Madina, F, A, Lapoz, *J. Alloys. Comp.* **2002**, 335,203.
- [29] Que W, Zhou Y, Lam YL, Chan YC, Kam CH, *Thin Solid Films*, **2000**,358, 4407
- [30] Rao AV, Kalesh RR, Pajonk GM, *J. Mater. Sci.*, **2003**, 38, 4407
- [31] Packter A and Oman A, *Cryst. Res. Technol.*, **1984**, 19 ,467.
- [32] V. Ramaswamy, R.M. Vimalathithan and V. Ponnusamy., *Pelagia Research Library-Advances in Applied Science Research*, **2010**, 1 (3): 197-204