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Synthesis and Characterization of Pure NiO Nanoparticles

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Abstract:

Nickel oxide (NiO) nanoparticles with a particle size of around 24 nm have been synthesized via chemical precipitation method. The synthesized precursor was dried and calcined at 300 K for 3 hours to obtain the NiO nanoparticles. The X-Ray diffraction analysis results indicated that the calcinated sample has cubic structure without any impurity phases. The FTIR analysis results confirmed the formation of NiO. Photoluminescence studies and SEM analysis are carried out. NiO nanoparticles formed with high purity.

Keywords: FTIR, SEM, Nanoparticle, NickelOxide, Photoluminescence

1. Introduction

Over recent years, the nano science have been an exciting area of research as much of application point of view as of fundamental research. These materials exhibit significant changes in their mechanical, magnetic, electric properties compared with its counterpart material in bulk form. NiO is a transition metal oxide and NiO nanoparticle have recently been the subject of renewed attention for presenting unusual features., mainly below 10 nm. Due to its novel properties NiO can be used as a catalyst, battery electrode, gas sensors, electrochromic films, photo electronic devices and so on[1].

The Structural property of NiO nanoparticles is closely related to the preparation techniques. Several methods have been used and developed for synthesizing NiO nanomaterials. To reduce the cost of chemical synthesis and to produce materials for technological applications, in this work Sol- gel method is used to prepare the NiO nanoparticles[2]. Here we synthesize the NiO nanoparticles and their structural, optical properties are studied.

2. Experimental Details

NiO nanoparticles were synthesized by a Sol-gel method. This process involves the formation of colloidal suspension (sol) and gelation of sol to form a network in a continuous liquid phase(gel). Initially Ni(OH)₂ solution was prepared by reacting aqueous solutions of 0.1 M of Ni(NO₃)₂·6H₂O and 0.5M of NaOH. To prepare this NaOH solution is added dropwise in to the Nickel nitrate hexa Hydrate solution with constant stirring until the pH of the system reaches to 12. The resulting green gel is washed several times with distilled water and acetone to remove impurities. The final product is dried in air and calcined for 3 hours at 300°C. The same procedure was repeated by using KOH instead of NaOH and the product named as sample2. The synthesized nanoparticles were characterized by X-ray Diffractometer, Fourier Transform Infrared Spectroscopy. SEM analysis and photoluminescence studies are also carried out.

3. Result and Discussion

3.1. XRD Analysis

The crystalline structure of nano NiO was determined by XRD analysis. The existence of strong and sharp peaks located at the 2θ values 37.16°, 43.29°, 62.8° corresponding to (1 1 1), (2 0 0), (2 2 0) planes respectively indicates the formation of pure NiO nanoparticles [Figure.1][3] The diffraction peaks of NiO can be matched with JCPDS data.[JCPDS file: 895881 & 652865]. The average particle size of the pure NiO nanoparticles was determined using the following Debye Scherrer equation and it was found to be 12.152 nm (sample 1) and 39 nm .

$$D = K\lambda / \beta \cos\theta$$

Where K is a constant equal to 0.39, β is the full width half maximum height of the diffraction peak at an angle θ and λ is the wavelength. Table I and II indicates the average particle size of the samples1 and 2 respectively.

Obs. Max.	Max. Int.(a.u)	FWHM	Particle Size
37.16	390	0.6	13.97
43.29	634	0.6	14.25
62.8	274	1	9.31
75.28	101	0.4	25.09

Table 1: Particle size of NiO nanoparticles (sample 1)

Obs. Max.	Max. Int.(a.u)	FWHM	Particle Size
43.17	0.7	16	12.2101
43.39	0.1	23	85.5361
62.74	0.6	82	15.5125

Table 2: Particle size of NiO nanoparticles (sample 2)

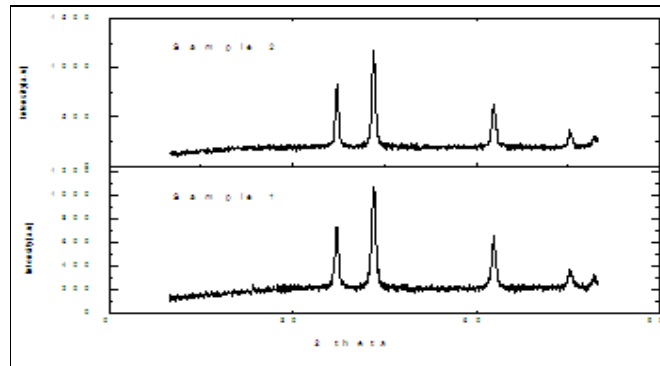


Figure 1: X-Ray Diffraction Pattern for Pure NiO nanoparticles

3.2. FTIR Analysis

In the FTIR spectra, the main bands were observed at ~ 3600,1632,1378,1030,830 and 414 cm^{-1} . The band at ~3600 represented stretching of O-H group. The Band 1632,1378 cm^{-1} represents carboxylate group of NiO. The band 1030 cm^{-1} represents C-O group in stretching mode. The band 830 cm^{-1} is the bending vibration of NiO [Figure.2]. In the FTIR spectra for sample 2, the main bands were observed at ~ 3448,1633,1383,825 and 428 cm^{-1} . The peaks are shifted towards higher frequency region.

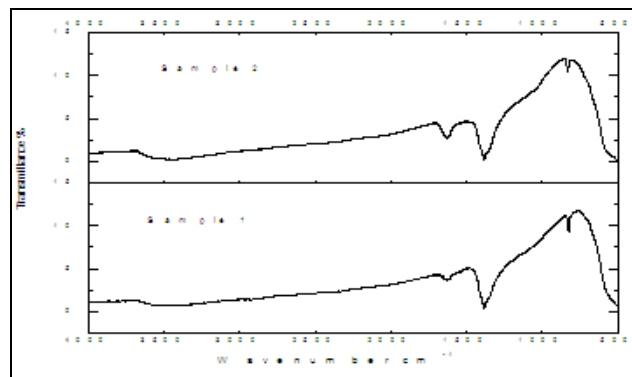


Figure 2

In the FTIR spectra for sample 2, the main bands were observed at ~ 3448,1633,1383,825 and 428 cm^{-1} . The peaks are shifted towards higher frequency region.

3.3. Photoluminescence Studies

Photoluminescence spectra of pure samples 1 and 2 [Figure.3] shows the origination of one main strong emission peak at 361 nm and many smaller emission peaks observed nearby 3755,392,410,440,492 nm. The origin of the main strong peak attributed to the electronic transition of the Ni^{2+} ions. The other peaks might be attributed to near band to band transition and oxygen related defects[4].

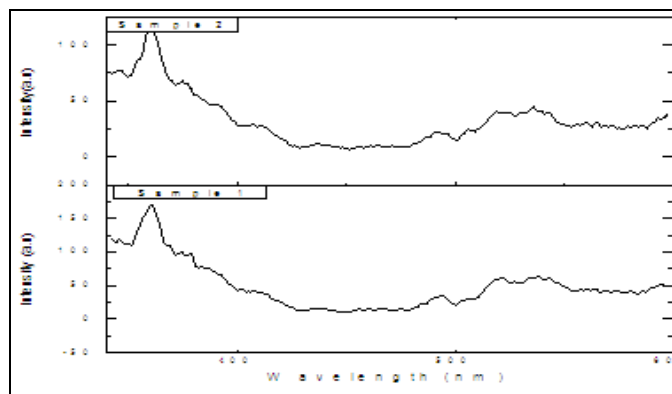


Figure 3: Photoluminescence spectra of pure NiO nanoparticles

3.3. SEM Analysis

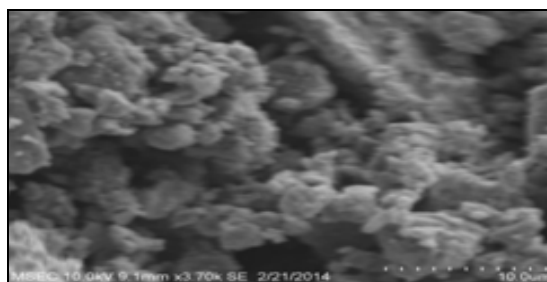


Figure 4: SEM images of pure NiO nanoparticles (Sample 1)

Scanning Electron Micrographs for pure NiO nanoparticles are shown in Figure-4. These results reveal that pure sample consists of nanoparticles with non homogeneous size and shape with average size around 100 nm. The SEM results are deviated from results for average size estimated by X-ray diffraction. The SEM micrograph shows agglomerated particles. The agglomeration can be induced by densification resulting from the narrow space between the particles due to the uniform distribution of oxidized metal anions in the three dimensional polymeric network structures. The nanoparticles tend to agglomerate during synthesis or delivery process due to their high surface area and surface energy.

4. Conclusion

NiO Nanoparticles were successfully synthesized by Sol-gel method XRD, FTIR; SEM studies are carried out for pure NiO nanoparticles. The particle size of synthesized NiO nanopowders is smaller for NiO around 25 nm. The FTIR spectra indicate the formation of Pure and Zn doped NiO nanoparticles. The XRD spectrum shows the samples are single phase and there is no impurity peak. The SEM & PL analysis also carried out. The results reveal that high purity NiO nanoparticles were prepared in this work. The pure NiO particles are having FCC structure.

5. References

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