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## Synthesis and characterization of nickel nanoparticles by sol-gel process

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### ABSTRACT

The Nickel nanoparticles were synthesized by using sol-gel process at 800°C. The structural features, chemical composition and optical properties of the nanoparticles were investigated by X-Ray Diffraction (XRD), X-Ray photoelectron spectroscopy (XPS) and Ultra Violet spectroscopy (UV) studies. The particle size was calculated by using Debye - Scherrer's Formula. The existence of fcc structure is confirmed by XRD technique.

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### KEYWORDS

Nickel;  
Nanoparticles;  
Scherrer's formula;  
Sol-gel process;  
Fcc.

### INTRODUCTION

In recent years, nanoparticles have attracted increasingly more attention year the materials community. Nanoparticles exhibit various shapes or forms and possess unique Chemical, Physical or Mechanical properties. The new class of microscopes has led to an amazing discovery: the vary properties of matter depends on its size. The nature of matter on a nanoscale is dramatically different from its bulk form. Its optical and electrical properties and even colors changes. New horizons of discovery, based on this feature, are emerging.

A nanometer is one billionth of a meter all three orders of magnitude smaller than a micron, roughly the size of a molecular itself (e.g. a DNA molecular is about 2.5 nm long while a sodium atom is about 0.2 nm)

The prefix "nano" means one billionth. One nanometer is 1/1,000,000,000 of a yard. Nanotechnology is an important field for the scientific and economical revival of the developing world.

A nanosystem is therefore something which suffi-

ciently small that we could not see with naked eye and not even with ordinary microscope<sup>[1]</sup>.

Nickel has recently received a great scientific interest because of its wide ranger of applications. A nanomaterial having considerable current interesting variety of areas and it exhibits improved physical, chemical properties, the selection of the materials with required properties for specific applications plays very important role. In this series, researchers are interested in studying the cheap and low cost materials<sup>[2]</sup>. As the size deceases to nanometer scale many of the mechanical, Electronic, Optical, Magnetic and Thermodynamic properties are significantly altered from those of either the bulk on the single molecule<sup>[3]</sup>.

Recently there have been many researches made attempt on synthesis of nickel nanoparticles, such as chemical vapour deposition<sup>[4]</sup>. Magnetron sputtering<sup>[5]</sup> spray paralysis<sup>[6]</sup>, growth techniques<sup>[7]</sup>, high temperature chemical route<sup>[8]</sup>, co-precipitations<sup>[9]</sup>, pulsed laser deposition<sup>[10]</sup>. Among the various technique the low temperature sol-gel process reveals more importance be-

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cause the final properties of the this films particles can be tailored using various precursors and different annealing temperature, stiochiometry of the final compound is homogeneity, minimizes air pollution and cost effective method<sup>[11]</sup>.

In the present study, the pure NiO and doped Ni<sub>0.8</sub>R<sub>0.2</sub>O (R = Mg /Zn) ie Ni<sub>0.8</sub>Mg<sub>0.2</sub>O and Ni<sub>0.8</sub>Zn<sub>0.2</sub>O nanoparticles have been successfully synthesized by a simple sol-gel process and the obtained product was characterized by XRD, XPS and UV measurements.

### EXPERIMENTAL DETAILS

#### Materials used

A.R. grade chemicals of purity 99.99% of Nickel Acetate [Ni(Ac)<sub>2</sub>. 4H<sub>2</sub>o], Magnesium Acetate [Mg(Ac)<sub>2</sub>. 4H<sub>2</sub>o] and Zinc Acetate [Zn(Ac)<sub>2</sub>. 4 H<sub>2</sub>o] were used as the precursor source materials for Ni, Mg and Zn respectively. 2- Methoxyethanol and monoethanamine are used as a solvent.

#### (A) Sol-gel preparation

The preparation of nanoparticles by sol-gel process consists of three stages viz., (a) preparation of the precursor solution, (b) drying at 150°C, (c) Annealing at 800°C.

#### (B) Preparation of the precursor solution

The precursor solution were prepared by dissolving the respective appropriate precursor source materials into the mixture (1:1 v/v) of 2-Methoxyethanol and monoethanamine. The solution was stirred at 60°C for an hour and then aged for 24 hours at room temperature.

#### (C) Drying and annealing

The powders were dried at 150°C for over night and then dried at 230°C for 5 hours. The dried powders are then annealed at 800°C for 5 hours to achieve the right phase.

### PHYSICAL MEASUREMENTS

Finally the powders were taken for the different characterization process such as X-Ray Diffraction, X-Ray Photoelectron spectroscopy and Optical measurements.

The XRD pattern of the nickel nanoparticles powders are collected from a X-Ray diffractometer (PW1710 diffractometer of Phillips) using Cu-K $\alpha$  radiations. The elemental compositional analysis has been carried out using XPS. The optical properties of the nickel nanoparticles were studied using UV-visible absorption (UV2450 spectrometer of SHIMADZU).

### RESULT AND DISCUSSIONS

The nickel nanoparticles synthesized by simple sol-gel method were characterized by XRD XPS and UV studies. Figure 1(a) shows the XRD pattern of pure NiO and Figure 1(b) shows the XRD patters of Ni<sub>0.8</sub>Mg<sub>0.2</sub>O NiO is anti-ferromagnetic material and has fcc structure. From the figure 1(a) and (b) it is clear that there is no considerable change in the position of the diffraction peaks with Ni<sub>0.8</sub>Mg<sub>0.2</sub>O indicating the existence of cubic structure. The XRD pattern was compared with JCPDS card No for NiO is 04-0835 and Ni<sub>0.8</sub>Mg<sub>0.2</sub>O is 34-0410. The XRD pattern was found to match perfectly with the standard data. It can be seen that both the nanoparticles which are processed at 800°C confirm it is still in cubic structure. The particle size (t) is calculated using the Scherrer's formula from full width at half maximum (FWHM)( $\beta$ )<sup>[12]</sup>

$$t = 0.94\lambda / \beta \cos\theta$$

Where,  $\lambda$  is wavelength of the X-Ray used,  $\beta$  is the FWHM in radian, t is the particle size, and  $\theta$  is half the angle between the incident and the scattered X-Ray beams. The average particle size was estimated to be 27.28 nm for pure NiO and 26.27 nm for Ni<sub>0.8</sub>Mg<sub>0.2</sub>O.

Figure 2(a) shows the XPS spectrum of pure NiO and figure 2(b) shows the XPS spectrum of Ni<sub>0.8</sub>Mg<sub>0.2</sub>O. From the figure 2(a) clearly indicates that the presence of Ni in the material. Similarly from the figure 2(b) presence of both Ni and Mg are noticed well. In addition to the precursor material, Carbon is also evidenced and this is due to the residual carbon<sup>[13]</sup> which is absorbed from the atmosphere, but it is not observed in the XRD pattern.

The UV-Visible absorption spectra of NiO, Ni<sub>0.8</sub>Mg<sub>0.2</sub>O and Ni<sub>0.8</sub>Zn<sub>0.2</sub>O annealed at 800°C is shown in figure 3(a). From the figure it is clearly indicates that the addition of Mg/Zn results the absorption edges of Ni<sub>0.8</sub>Mg<sub>0.2</sub>O and Ni<sub>0.8</sub>Zn<sub>0.2</sub>O are shifted from the ab-

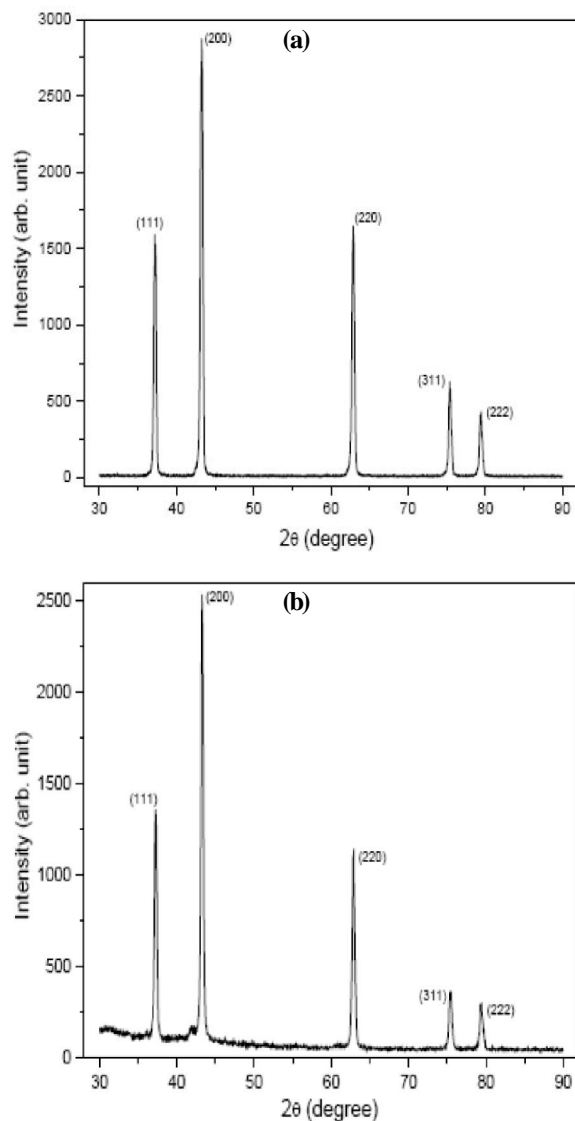


Figure 1 : (a) and (b) XRD pattern of pure NiO and  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$  annealed at  $800^\circ\text{C}$ .

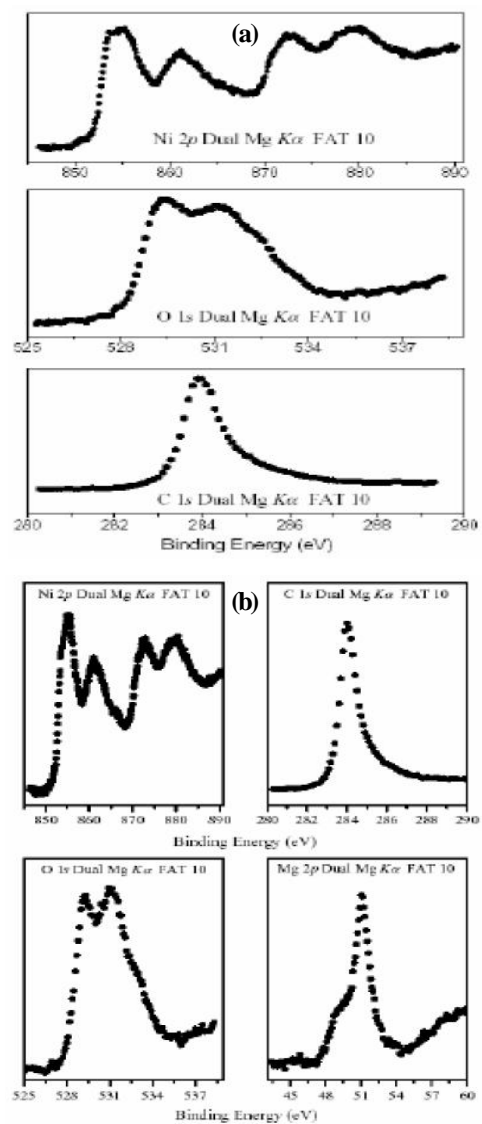


Figure 2 : (a) and (b) XPS spectra of pure NiO and  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$  annealed at  $800^\circ\text{C}$ .

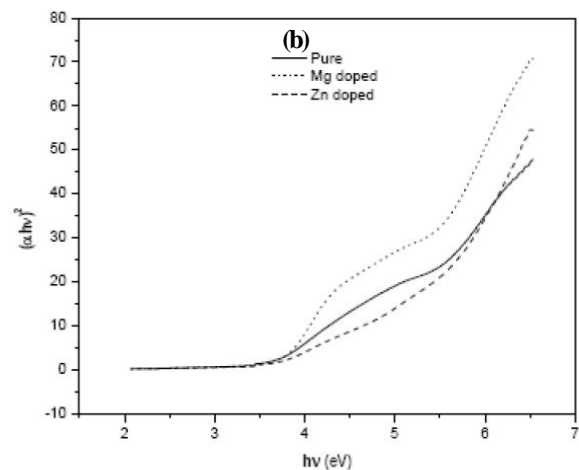
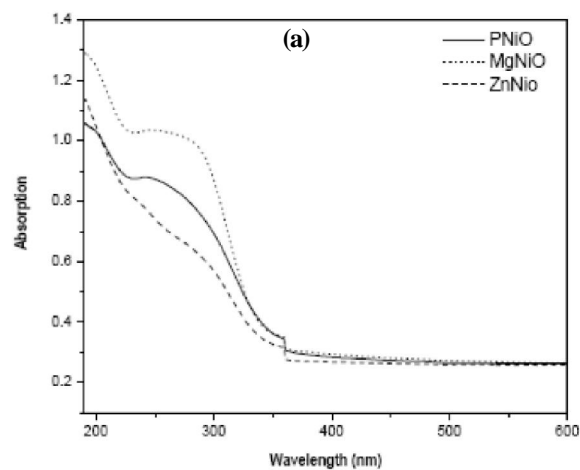


Figure 3 : (a) and (b) UV spectra of NiO,  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$  and  $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{O}$  and absorption coefficient  $(\alpha \cdot hv)^2$  versus photon energy ( $h\nu$ ) for NiO,  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$ .

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sorption edges of NiO. Absorption edges of NiO appears at  $\lambda=244\text{nm}$ , whereas the absorption edges for  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$  and  $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{O}$  appears higher (above) and shorter (below) the wavelength corresponding to NiO. The optical band gap values of the prepared nanoparticles were determined by plotting graph between  $(\alpha h\nu)^2$  versus  $(h\nu)$  is shown in figure 3(b). The band gap values of NiO is 3.6eV,  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$  is 3.75eV and  $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{O}$  is 3.571eV. From the observed band gap values indicates that the addition of MgO into pure NiO leads to increases the band gap of  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$ , while the addition of ZnO decreases the band gap of  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$ .

### CONCLUSION

The nanoparticles of nickel have been successfully synthesized by a simple sol-gel process. The existence of fcc structure is confirmed from the XRD analysis. The particle size was estimated to be 27.28nm for NiO and 26.27 nm for  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$ . The particle size was calculated from Debye Scherrer's formula. The presence of Ni and Mg are noticed well from the XPS analysis.

The band gap values of NiO is 3.6eV,  $\text{Ni}_{0.8}\text{Mg}_{0.2}\text{O}$  is 3.75eV and  $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{O}$  is 3.571eV was determined from the UV analysis.

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