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RESEARCH ARTICLE

Effect of Annealing on NiO Nanoparticles for the Study of Structural, Morphological & Optical Properties

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Abstract

In the present analysis, NiO nanoparticles have been synthesized by Sol-Gel Method. The effect of annealing temperature on NiO nanoparticles showed change in size, morphology and optical band gap. All the finding confirms the formation of pure NiO nanoparticles. X-Ray Diffraction (XRD) analysis of the samples shows the structure of the synthesised material is rhombohedral with R-3m space group. When the annealing temperature increased, the crystalline nature increased and size of the nanoparticles decreased. The Optical band gap of as prepared and annealed samples were calculated 2.8 eV and 3.25eV respectively. The presence of Ni-O nanoparticles was confirmed by Fourier Transform Infrared Spectroscopy (FTIR) analysis. From the morphological analysis it has been observed that the average size of nanoparticles is in the range of 35-250 nm and the particles are spherical in shape.

Keywords: NiO, Absorption, Annealing Temperature, Crystalline nature, Optical band gap.

Introduction

The science of nanomaterials evolved as frontier application of nanoparticles. This branch of science explains how to fabricate and analysis of nanomaterials. Nanomaterials fabrication is differentiate into three categories based on their dimensionality: zero dimensional, one dimensional, two and three dimensional. Nowadays, nanotechnology is used in areas such as Pharmaceuticals, Medicine, Electronics, Robotics, and Tissue engineering. NiO material shows versatile properties like mechanical, electronic, magnetic, thermal, catalytic and optical as its applications are electrochromic films, fuel cell electrodes and gas sensors. Heavy metal removal from wastewater has been intensively explored in recent decades using methods such chemical precipitation, electrochemical processes, membrane filtration and adsorption. NiO nanostructures showed excellent magnetic behaviours such as superparamagnetic, ferromagnetic and antiferromagnetic order depending on the shape & size. For the synthesis of nanomaterials there are various methods to fabricate like sol gel method, hydrothermal synthesis, precipitation calcination method, flame spray pyrolysis etc. We have prepared our samples by using Sol Gel Method. The ability to create homogenous nanostructures at low temperatures and great purity were the main benefits of the sol-gel manufacturing technology. In this liquid phase synthesis, the dispersion can be stabilized by capping the particles with the suitable ligands (Ying *et al.*, 2007). The sol-gel technology aims to regulate a material's dimensions on a nanoscale scale from the very beginning of production. Materials' properties can be improved through chemical processing, controlled high purity, and enhanced uniformity. This lower temperature processing method has many benefits over the more traditional methods of synthesizing nanoparticles. Fiber pulling, film coating, and net form casting are further benefits of this method (Choi and Ben-Nissan *et al.*, 2014). Now a present point of view is focussed on a novel synthesis technique to synthesize NiO nanoparticles using sol-gel technique by using $NiCl_2 \cdot 6H_2O$ as the inorganic precursor. This process involves dissolving the molecular precursor (typically metal alkoxide) in water or alcohol, heating it, and stirring it until it gels. According to the gel's intended usage and desirable characteristics, it must be dried suitably because the gel produced during the hydrolysis/alcohololysis method is wet or moist. (Ciobanu *et al.*, 2013). For instance, if the liquid is alcoholic, burning alcohol is utilised to finish the drying process. After the drying phase, the produced gels are

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crushed and then calcined. (Karthik *et al.*, 2011). Because of the low processing temperature and cost-effectiveness of the sol-gel process, it is possible to effectively regulate the products chemical composition. (Khan *et al.*, 2011). The samples were characterized and analyzed with the influence of experimental parameters, such as calcination temperatures, PH level and concentration of nanoparticles by different Characterization techniques.

Materials and Method

Materials: The chemicals used in the synthesis were $NiCl_2 \cdot 6H_2O$ & NaOH. All the solutions were prepared in Ethanol.

Synthesis of NiO nanoparticles

One solution of 1.5gm of $NiCl_2.6H_2O$ and 70 ml absolute ethanol was added in the another solution of 0.5 gm NaOH dissolved in 100 ml absolute ethanol in a beaker at room temperature. The Solution was subjected to continuous stirring. It was stirred for 2 hours. After this we get light green coloured Gel. Then the gel was filtered and washed many times with water and Ethanol. Fine green powder was subjected to Calcination at 500°C for 30 minutes. After this black coloured nanopowder of NiO were produced. The flow chart for the same is shown in Fig. 1.

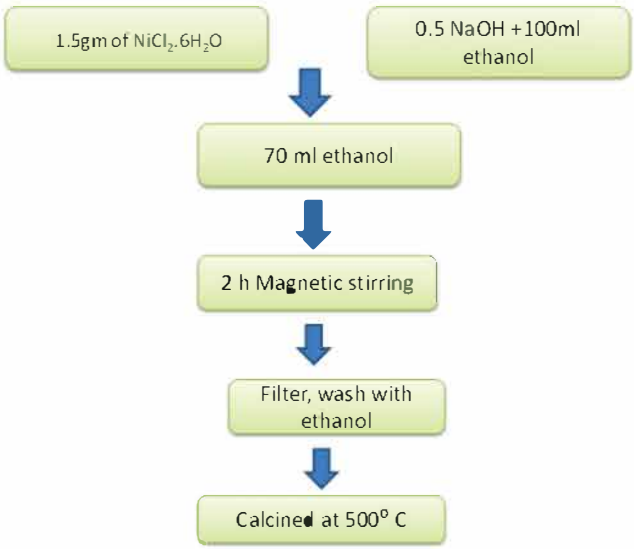

Fig.1. Flow chart for the synthesis of NiO nanoparticles by Sol Gel Method

Characterization

The X-ray powder diffraction patterns of the NiO Nanostructure fabricated by sol-gel method analyzed with Bruker D8 advance X-ray diffractrometer using CuKα radiations scanned from 20° to 90°. Scanning Electron Microscopy (SEM) and Energy Dispersive Analysis X-ray Spectroscopy (EDAX) investigated the surface morphology and elemental composition of the fabricated samples. The Composition and Chemical bonds were analyzed by FTIR in the wavelength range 400-4000 cm^{-1}. UV-Vis Spectroscopy studied absorption spectrum & optical band gap of the material in the range of 200-800 cm^{-1}.

Result and discussion

Structural Analysis

All peaks of NiO samples synthesized by the sol-gel method correspond precisely to the JCPDS data (014-0481) is shown in Fig. 2. It was crystalline, with 2θ peaks recorded at 2θ=43.2° (200), 2θ=51.5° (112), 2θ=56.7° (202), 2θ=62.7° (220), 2θ=66.1° (004), and 2θ=75.2° (015). By comparing the XRD patterns with JCPDS data, it was confirmed that the material is nickel oxide. The nickel oxide nanoparticles exhibit a rhombohedral structure with R-3m space group and a = 2.94nm and c = 7.23nm lattice constants (Jeevanandam and Pulimi, 2012). The average crystallite size (D) of nanoparticles obtained by Debye-Scherrer's formula given by equation

$$D = K\lambda / (\beta \cos\theta) \qquad (1)$$

Where D is the crystallite size; λ is the wavelength of the X-ray radiation (λ=0.15409 nm) for CuKα; Uncalcined sample formed by the Sol-Gel method was amorphous. After strong annealing, crystalline nanoparticles were prepared and peaks are shifted with the annealing temperature analyzed by the XRD technique (Ashraf and Khan, 2015). According to the XRD pattern the average size of the as-prepared NiO nanostructure was 56 nm and 32 nm annealed at 500°C as calculated by Debye-Scherrer's formula.

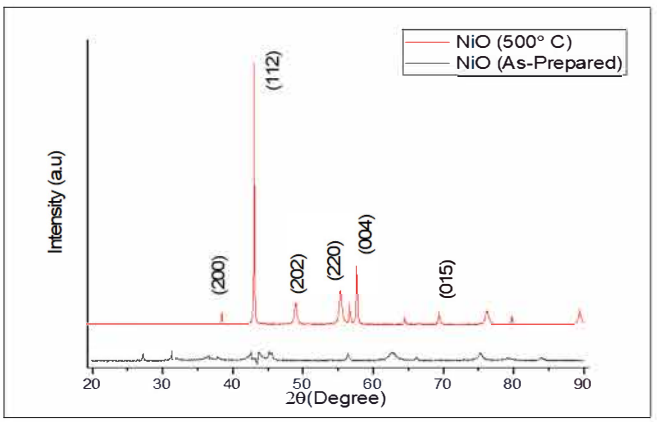

Fig.2. XRD Pattern of NiO nanoparticles

Morphological Analysis

The surface morphology of pure and annealed NiO nanoparticles was taken at different magnification by

SEM shown in Fig. 3. The images show that nanoparticles are spherical in shape and they are polycrystalline. The scan revealed that particles formed clusters. The measured grain size exceeds the calculated crystallite size based on XRD measurements. The result revealed that highly crystalline and monodispersed nickel nanoparticles were produced. Nickel nanoparticles tend to aggregate due to their high surface energy and surface tension, which may explain the discovery of certain larger nanoparticles (Rahdar *et al.*, 2015). According to the qualitative elemental analysis shown by the EDAX spectrum is shown in Fig. 4, only nickel and oxygen peaks are present, with no other peaks discernible. We can therefore conclude that pure crystalline NiO nanoparticles were synthesized.

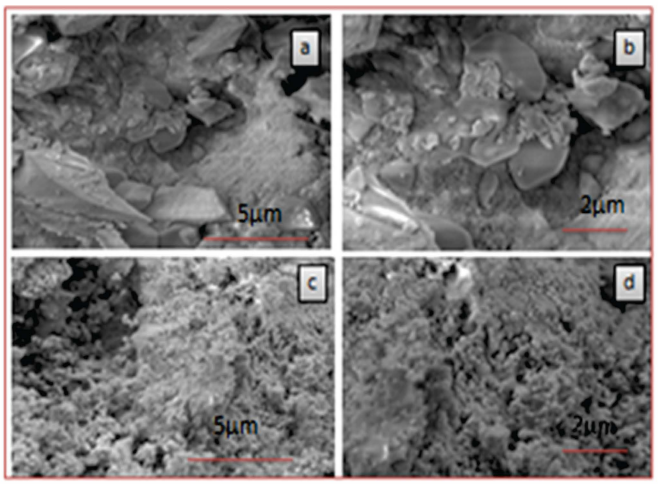

Fig.3. SEM image of (a-b) as prepared NiO (c-d) Annealed NiO 500 °C nanoparticles at different magnification

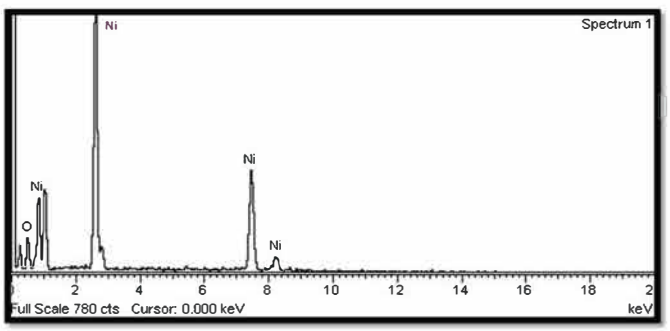

Fig.4. EDAX of pure NiO annealed at 500°C

FTIR Spectroscopy

FTIR analysis is used to discover the chemical bonds that are present in the material. The preparation of the samples involved combining the powdered materials with KBr, which were then ground and pressed into a transparent pellet with a diameter of cm^{-1}. The absorption band at 3641 cm^{-1} on the FTIR spectrum of uncalcined NiO sample is represented by O-H bond shown in Fig.5 (a). Our samples were prepared in water, therefore the presence of hydroxyl groups on their surfaces is to be expected. The absorption band at 1638 cm^{-1} represents hydroxyl groups. Ni-O vibrational bond is responsible for absorption bonds 465 and 513 cm^{-1}, while a Ni-O-H stretching bond accounts for an absorption bond 637 cm^{-1}. The peak at 2370 cm^{-1} is due to the C-H vibrational mode. The frequency of 1469 cm^{-1} corresponds to C-H deformation. The band at 1722 cm^{-1} indicates the presence of C=O stretching in a carbonyl group that is uncoordinated and unionized. The 1621 cm^{-1} band corresponds to the H-O-H bending mode, confirming the presence of water molecules. The rise in the range between 1000 and 1100 cm^{-1} corresponds to the C-O stretching. The band of absorption that can be seen at 3441, 3434 and 3424 cm^{-1} on the FTIR spectrum of annealed NiO samples at 500^0C represented to O-H bond shown in Fig. 5(b) (Rahdar *et al.*, 2015). The absorption band at 1374 and 1441 cm^{-1} are present due to hydroxyl groups. The observed absorption bonds at 378, 398, 417, 426 and 456 cm^{-1} showing the Ni-O vibration bonds, and bonds at 832 cm^{-1} showing Ni-O-H stretching bond respectively. Some vibration bonds are observed might be due to stretching vibrations of organic impurity. The cation mass, cation-oxygen separation, and bonding force all affect the vibrational frequencies. (Ashraf and Khan, 2015).

Optical Analysis

The UV-Visible absorption spectrum of NiO nanoparticles obtained for pure and annealed shown in Fig.6.The optical absorption coefficient α of a semiconductor is expressed by the following equation:

$$\alpha = A(h\nu - E_g)^n / h\nu \qquad (2)$$

Here, α is the absorption coefficient, E_g is the absorption band gap, A is a constant depending on the transition probability, n depends on the nature of the transition (Tauc, 1968). In Fig. 6 (a) the presence of NiO nanoparticles was confirmed by a significant peak, which is an absorption peak in between the range of 200-400 nm. This observed blue shift is due to a decrease in the size of nanoparticles (Boschloo *et al.*, 2001). In Fig. 6 (b) from the tauc plot, the observed values of band gap are 2.8eV and 3.25eV for NiO nanoparticles as-prepared and annealed respectively. As increase the annealing temperature the size decreases and the optical band gap increases. It can be concluded that the band gap of the prepared material have been found to be particle size

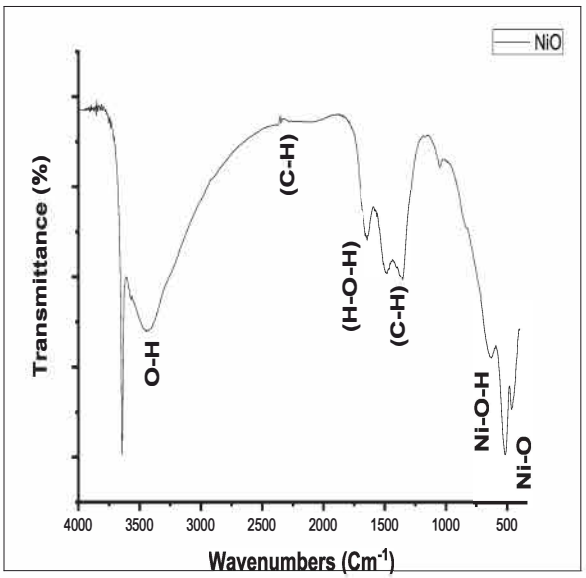

Fig.5 (a). FTIR spectrum of pure NiO

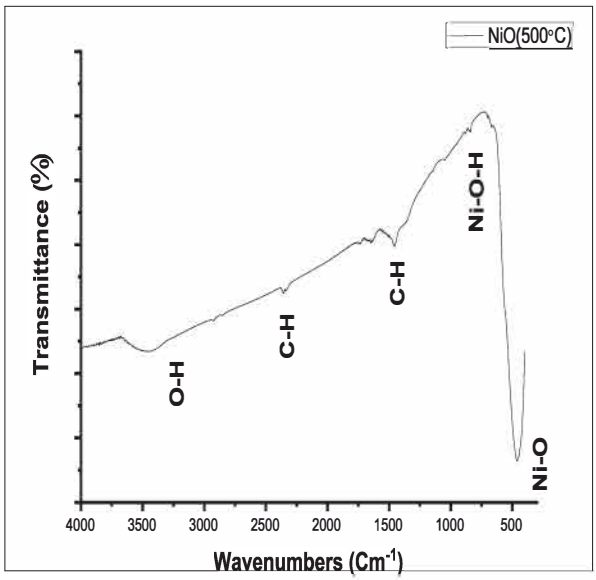

Fig.5 (b). FTIR spectrum of annealed NiO(500°C)

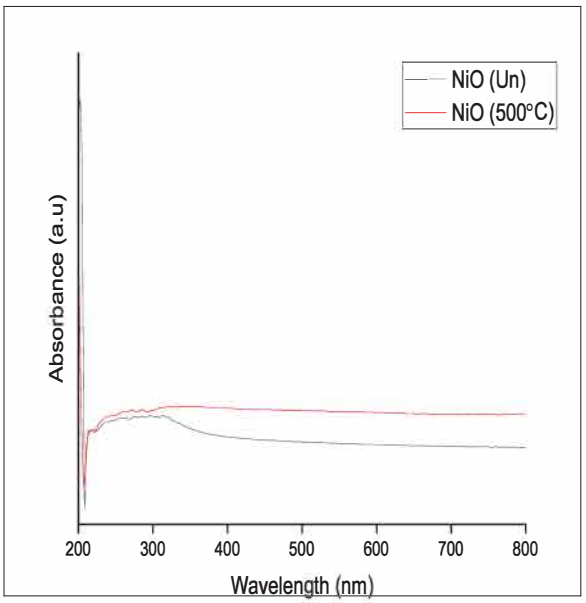

Fig.6(a). Absorption spectra of pure NiO (as prepared & 500°C)

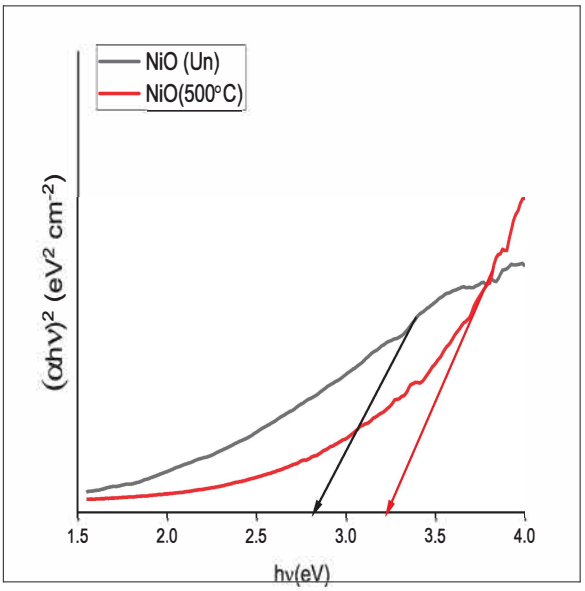

Fig.6 (b). Bandgap of NiO (as prepared & 500°C)

dependent (Anandan and Rajendran 2011; Faranak and Negar, 2015).

Conclusion

In this investigation, NiO is synthesized through the chemical method i.e. sol-gel method. The prepared samples of NiO were analysed by XRD, UV-Vis Spectroscopy, FTIR, EDAX and SEM. XRD analysis of the samples showed crystalline nature with annealing temperature. The nickel oxide nanoparticles exhibit rhombohedral structure with R-3m space group. The crystallite size is calculated for as prepared and annealed samples are 56 nm and 32 nm respectively. The UV-Vis spectrum showed an increasing absorption edge due to the annealing temperature in UV region for NiO nanoparticles. The band gap of the as prepared NiO is 2.8 eV and annealed at temperature 500ºC is found 3.25eV. FTIR analysis confirms the presence of Ni-O nanoparticles. From SEM analysis the morphological features of prepared nanoparticles were observed of average size in between 35 - 250 nm which were spherical

in shape and presence of elementary component confirmed by EDAX.

Acknowledgement

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