

RESEARCH ARTICLE

Exploring the Characteristics of the Nickel Oxide Nanoparticles via Sol-gel Method

M. Jothi^{*1}, K. Sowmiya¹

ABSTRACT

Nickel Oxide (NiO) is an important transition metal oxide with cubic lattice structure. NiO is thermally stable that is suitable for tremendous applications in the field of optic, ceramic, glass, electro-chromic coatings, plastics, textiles, nanowires, nanofibers, electronics, energy technology, bio-medicine, magnetism and so on. In this present study, NiO nanoparticles were successfully synthesized by sol-gel technique. Nano-sols were prepared by dissolving Nickel-Chloride [NiCl₂.6H₂O] in NaOH solvent and were converted into nano structured gel on precipitation. A systematic change in preparation parameters like calcination temperature, time, pH value has been noticed in order to predict the influence on crystallite size. Then the prepared samples were characterized by the X-ray Diffraction Spectroscopic (XRD), UV-VIS Spectroscopy, Fourier Transform Infra-Red Spectroscopy (FTIR), Energy Dispersive X-ray Spectroscopy (EDX), Scanning Electron Microscopy (SEM) and Particle Size Analyzer (PSA). From XRD, the average crystalline-size has been calculated by Debye-Scherrer Equation and it was found to be 12.17 nm and the band gap energy of Nickel oxide (NiO) from UV studies reveals around 3.85 eV. Further, EDX and FTIR studies, confirm the presences of NiO nanoparticles. The SEM study exhibits the spherical like morphology of Nickel oxide (NiO). Further from PSA, the mean value of NiO nanoparticles has been determined.

Keywords: Nano-sols, Sol-gel, XRD, FTIR, EDX.

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1. INTRODUCTION

Nanotechnology is a scientific approach that includes the synthesis of nano structured materials ranging from 1 to 100nm.^[1] Further, the preparation of inorganic and chemical metal oxide nano-particles has gained concentration for the past few decades, in physical, chemical, biological, medical, optical, mechanical and engineering sciences, to explore and handle single atom and molecules.^[2] Thus, the nanomaterials, so far discovered were well in economic circulation which leads to the invention of many new products.^[3,4] To ignore the use of poisonous chemical solutions which lead to various dangerous reactions and to avoid such reactions, conditions such as temperature, pressure and time, have been investigated by introducing the prospect of manufacturing nano- products in aqueous medium with the help of capping agents.^[5] In the present work, NiO has been synthesized using sol-gel technique. As Nickel Oxide (NiO) nanomaterials are quite good, cheap and are very stable. The use of nickel oxide is to prevent the nickel deficiency which has the interesting and essential applications in different

fields. It is recommended that recycling of nickel is a value-added economic activity that supports communities and governments, so the synthesis of nickel oxide is very essential.

Further, NiO is a transition metal oxide and p-type semiconductor which possess a wide energy gap (3.6 to 4) eV.^[6] Hence the number of researches on synthesis of Nickel Oxide nanoparticles is increased in these recent years due to their magnetic properties and potential uses in many fields including catalysis, memory - storage devices and sensors, also used in medical applications such as magnetically controlled drugs delivery, magnetic resonance imaging and hyperthermia treatment of cancer cells and so on.^[7-9] It's also plays a vital role in the fabrication of carbon-nanotubes (CNTs),^[10] and also exhibits potential applications including electronics, magnetism, Energy-technology and Bio-medicine.^[11-13] Due to their high reactivity, operational simplicity and eco-friendly properties, they are used to catalyze various organic reactions including chemo selective oxidative coupling of thiols, reduction of aldehydes and ketones, hydrogenation of olefins, synthesis of stilbenes

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from alcohol through witting type definition, i.e., by the strategy of simultaneous generation of ylides and aldehydes, alcohols are directly transferred to olefins with no need of preparation of either aldehydes or ylides, and α -alkylation of methyl ketone.^[14-18] They also catalyze certain inorganic reactions like decomposition of ammonia.^[19] Nano-crystalline NiO powder shows super-Para magnetism effects.^[20] These applications can be enhanced by decreasing the particle size in Nanoscale. Thus, nanoparticles with smaller size will also shows good anti-bacterial activity.^[21]

Various synthesis techniques have been applied to prepare Nickel Oxide nanoparticles such as Hydrothermal, Solid-state reaction, Electro-chemistry, Micro-emulsions Spray-pyrolysis, Precipitation method and Co-precipitation method.^[22-29] Among these sol-gel method gains more advantages over the other methods, as it requires relatively low temperature, can create very fine powder and produces composition not possible by solid-state fusion, low cost and simple technique. Hence, the Sol-Gel process has been employed for the synthesis of "Nickel Oxide" (NiO) nanoparticles. In this process, the solution evolves gradually towards the formation of a gel like network containing both a liquid-phase and a solid-phase.^[30] Typical precursors are metal-alkoxides and metal-chlorides, which undergo hydrolysis and poly condensation reactions to form a colloid. The basic structure or morphology of the solid-phase can range anywhere from discrete colloidal particles to continuous chain-like polymer networks.^[31] The applications are electrophoresis,^[32] inkjet^[33] and their medical applications are Wound-healer,^[34] Scar-size decrease,^[35] and Thrombosis treatment is possible by developing a new family of "injectable composites plasminogen" activator entrapped within alumina.

2. EXPERIMENTAL PROCEDURE

Nickel-Oxide nano powder is prepared by dissolving 3 grams of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 70 ml of the distilled water. It was allowed for stirring in the magnetic stirrer for 1 hour. Further, the solution of NaOH is prepared by dissolving 1.0 grams of NaOH pellets in 100 ml of distilled water then the prepared solutions was added drop wise ($\text{NiCl}_2 + \text{NaOH}$) and stirred for 2 hours. After stirring, light green gel was formed and kept in room temperature then, filtered and washed with water and ethanol. Thus, the "Light green colored precipitate" was obtained, dried at oven in 100°C for 5 hours leads to light green color powder which was calcinated at 300°C for 2 hours in the muffle furnace. Finally, the green color powder was

changed into black colored nano powder. Thus, the black colored Nickel Oxide (NiO) nanoparticle was prepared.



The prepared samples were characterized by UV-Vis Absorption, Fourier Transform Infrared (FTIR), X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), Particle Size Analyzer (PSA), and Energy Dispersive X-ray Spectroscopy (EDX).

3. RESULTS AND DISCUSSION

3.1 X-ray diffraction

The X-Ray Diffraction is to measure of the average spacing between layers or rows of atoms and to determine the orientation of a single crystal, size, shape and internal stress of small crystalline regions. The XRD pattern of the Nickel oxide (NiO) nanoparticles after calcination is shown in Fig. 1. This pattern exhibits sharpened reflection peaks which indicates that the crystalline size of NiO nanoparticles. The positions of peaks occurred at $2\theta = 37.2611, 43.2826, 62.8354$ has been indexed at (111), (200), (220) crystal planes of the NiO respectively,^[36] well match with the reported values.^[37] The sharpness and the intensity of the peaks indicate the well crystalline size of the NiO sample. Further, the grain-size was calculated by Debye-Scherrer Equation. Table 1 shows the various structural parameters that were calculated from the obtained XRD pattern. The average crystalline size of the Nickel oxide nanoparticles has been found to be 12.17 nm.

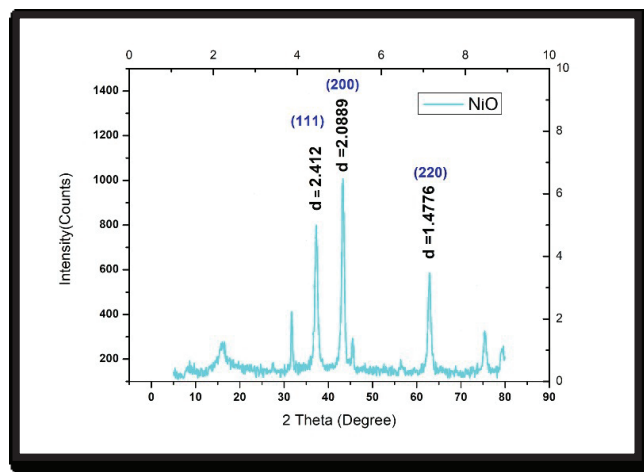


Fig. 1: XRD pattern for nickel-oxide nanoparticles

Table 1: Structural parameters of NiO nanoparticles

S. No	2° (deg)	Interplanar-distances (d)	Crystalline- Size ($D \cdot 10^{-8}\text{m}$)	Dislocation-Density ($\delta \cdot 10^{15}$)	Micro- Strain ($\epsilon \cdot 10^3$)
1.	37.2611	2.4122	1.17157	7.2855	3.0902
2.	43.2826	2.0889	1.22938	6.6164	2.9448
3.	62.8354	1.4776	1.25084	6.3913	2.8944

3.2 Scanning Electron Microscope (SEM) Analysis

The SEM is used to determine the particle size and the surface morphology of the synthesized nanoparticles by using a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. Typical SEM images of the Nickel oxide (NiO) nanoparticles were shown in Fig. 3.2, which shows the morphology of the synthesized powder at different magnifications of 55,000 with particle sizes. The results indicate that particles are in spherical shaped nano clusters with agglomeration are formed.^[39]

3.3 Energy Dispersive X-Ray Analysis (EDX)

The composition of NiO nano powder was examined by the Energy Dispersive X-ray Spectroscopy (EDX), a micro analytical technique used in association with SEM.^[40] The EDX examine the X-ray emitted from the synthesized sample, when electrons bombarded on the surface of the sample. By measuring the intensity and energy of the signal, data about the chemical composition was detected. The EDX spectrum shows frequency of X-rays in count of each energy-level. The peak's intensity determines the amount of the element in the sample. Since the EDX, demonstrated that explored NiO nano-powders consists of 53.43 % of Ni-content and

46.57% of O-content without the presence of any other type of elements in Fig. 3.

3.4 Fourier Transform Infrared Spectroscopy (FTIR) analysis

FTIR is a technique used to determine quantitative and qualitative features of IR-active molecules in organic or inorganic solid, liquid or gas samples.^[41] The FTIR was employed to identify the functional groups of the synthesized material. Fig. 4 shows the FTIR Spectra of explored Nickel oxide nanoparticles prepared by sol – gel method. The peaks at 3828.70 cm^{-1} and 3531.66 cm^{-1} represent the stretching of [O–H] band. The Alkyne group [$\text{C}\equiv\text{C}$ and $\text{C}\equiv\text{N}$] observed at 2358.94 cm^{-1} . The peak 1423.47 cm^{-1} corresponds to the bending of C–H Alkane group. The bending [$\text{C}=\text{H}$] Alkene group peak observed at 991.41 cm^{-1} .^[42]

The characteristic absorptions functional groups and type of vibrations with intensity were listed in the Table 2.

3.5 Ultra-Violet Visible Spectroscopy studies

UV Spectroscopy refers to absorption spectroscopy or Reflectance spectroscopy in the ultra-violet region and visible region. UV- VIS spectroscopy is commonly preferred technique for estimating the optical properties of the prepared samples. It states that the absorbance of

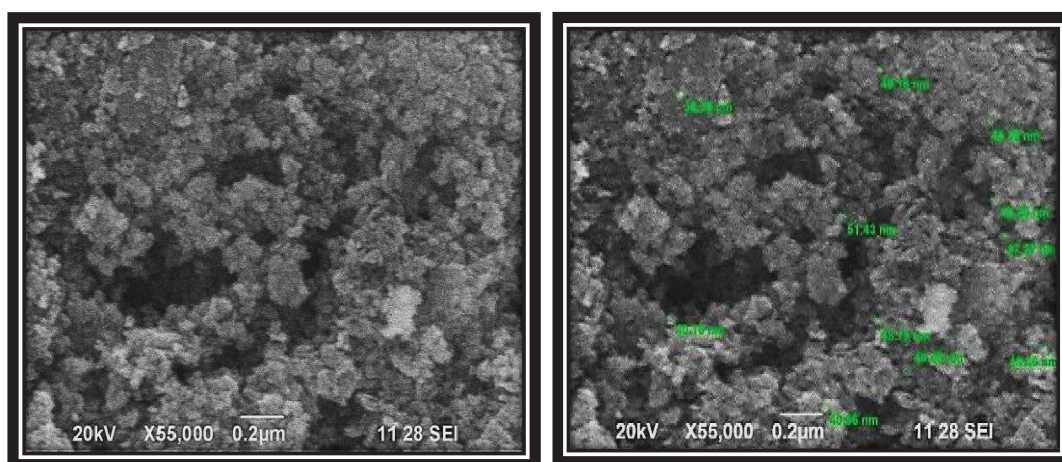


Fig. 2: SEM images of NiO nanoparticles

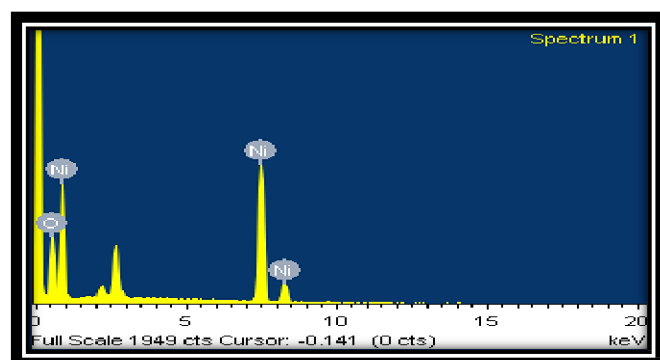


Fig. 3: EDX spectrum of NiO nanoparticles

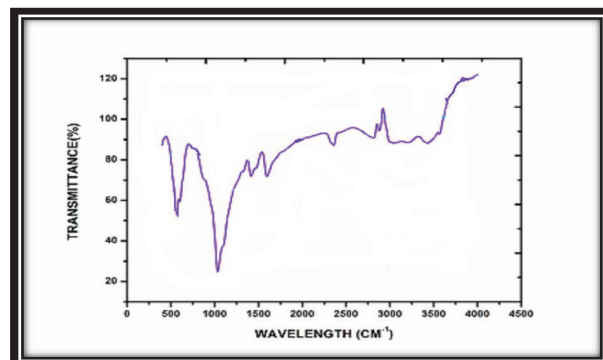
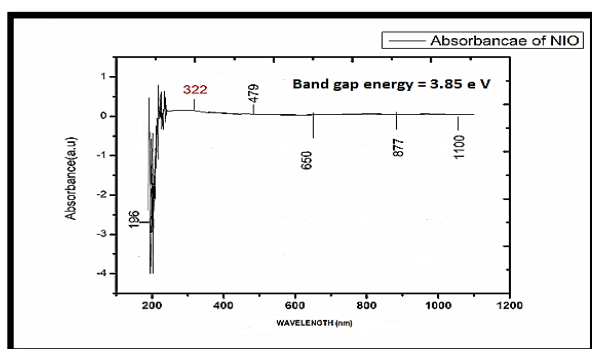
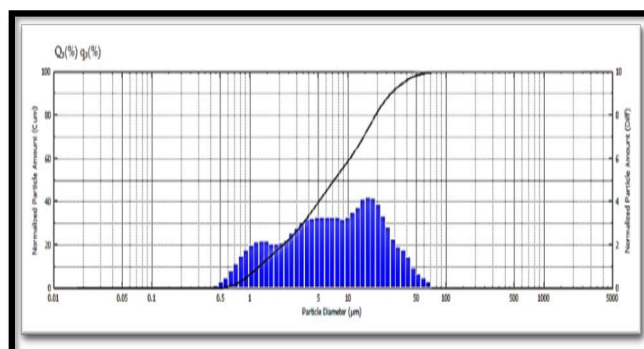


Fig. 4: FTIR spectrum of NiO nanoparticles

Table 2: Functional groups in IR spectra of NiO

Functional- Groups	Wave number (cm ⁻¹)	Type of Vibrations	Intensity
Alcohol O-H	3828.703531.66	Stretch, Free	Strong, Sharp
Alkyne C≡C and C≡N	2358.94	Stretch	Variable, not present in symmetrical
Alkane -C-H	1423.47	Bending	Variable
Alkene =C-H	991.41	Bending	Strong

**Fig. 5:** UV visible spectra of NiO nanoparticles**Fig. 6:** Distributions of NiO nanoparticles**Table 3:** Particle size distributions with diameter

Distribution Q (%)	Diameter X (μm)
25 %	2.725
50%	7.125
75%	16.368

a solution is directly proportional to the concentration of the absorbing species in the path length. UV Visible absorption spectra of Nickel oxide (NiO) are shown in Fig: 3.5, that ranges from 200 to 1200 nm. The absorption spectra of NiO is observed at 322 nm. Thus, the calculated band gap energy found to be 3.85 eV well matched with the reported value.^[6]

36 Particle Size Analyzer (PSA) studies

The Particle Size Analyzer (PSA) is the collective name of the laboratory techniques that examines the size range, and the mean size of the particles in a powder sample. The device which is used to measure the particle size is known as Particle Size Analyzer (PSA). The PSA is used to find the mean crystalline size of the synthesized NiO nanoparticles. The material as dispersed completely in distilled water. The Mean crystalline size of NiO nanoparticles was found to be 7.125 μm. The percentage of particle – distribution with diameter of NiO nanoparticles are given in the Table 3.

The particle size distribution graph of the NiO nanoparticles is shown in Fig. 6.

4. CONCLUSION

Nanoparticles of Nickel oxide (NiO) have been successfully explored by low temperature sol gel method. The XRD pattern indicates nano crystalline size of the particles that was found to be 12.17 nm. The SEM image exhibits NiO nanoparticles with spherical shape morphology in nanoscale. The EDX indicates the compositions of NiO nanoparticles i.e., 53.43% of Ni-content and 46.57% of O-content. The FTIR shows the corresponding functional groups of synthesized NiO nanoparticles. The PSA reveals the average particle size of NiO nanoparticles and found to be 7.125 μm.

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