SYNTHESIS OF NIO NANOPARTICLES BY CO-PRECIPITATION METHOD

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Abstract :

Nanostructured nickel oxide (NiO) nanoparticles were successfully synthesized via a co-precipitation technique using nickel(II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$) as the nickel precursor and sodium hydroxide (NaOH) as the precipitating agent. The structural characteristics and chemical composition of the prepared NiO nanoparticles were systematically investigated using X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy. XRD analysis confirmed the formation of single-phase NiO with a face-centered cubic (FCC) crystal structure and a high degree of crystallinity. The average crystallite size, estimated using the Scherrer equation, was approximately 18 nm. FTIR spectra further verified the presence of characteristic Ni–O vibrational modes, confirming the successful formation of NiO nanostructures. The results indicate that the co-precipitation method provides a simple and effective route for producing highly crystalline NiO nanoparticles suitable for potential functional applications

Keywords : Nickel nanostructures, Electrical properties

Introduction :

Nanoscience and nanotechnology are the study and application of extremely small things and can be used across all the other science field, such as chemistry, biology, physics, material science and engineering. Nickle oxide is a prominent p-type semiconductor with a wide band gap of approximately 3.6-4.0 eV, high thermal and chemical stability, and excellent redox beheavior. These properties make NiO nanoparticles suitable for applications in supercapacitors, batteries, gas sensors, photocatalysts, and spintronic devices. The nanoparticles also exhibit a rock-salt (NaCl-type) crystal structure, where each Ni^{2+} ion is octahedrally coordinated by six O^{2-} ions, providing a stable framework for electronic conduction and catalytic reactions.[1]

The Co-precipitation method is a simple and cost-effective technique used to synthesize metal oxide nanoparticles. It is the wildly used synthesis technique for synthesis NiO nanoparticles. In the present work we have synthesis of NiO NPs by coprecipitation method.[2]

Synthesis method :

1. Materials :

Nickel nitrate hexahydrate Ni (NO3)2.6H2O (98% extra pure) MW=290.80, Loba chemie Pvt. Ltd. and sodium hydroxide (Pellets 97% extra pure) MW=40.00. The solvent used for all mentioned chemicals was distilled water. All the synthesis procedure was cried out at room temperature.

2. Synthesis of NiO nanoparticles :

Nickle oxide nanoparticles were obtained by chemical co- precipitation method. 1 M solution of nickel nitrate were prepared by dissolving 14.54 g of $Ni(NO_3)_2{\cdot}6H_2O$ in 50 mL of distilled water with continuous stirring for 20 minutes to ensure complete dissolution. To this solution, 0.1 M NaOH were added gradually using a dropwise method to adjust the pH to 10. The mixture were then stirred at room temperature for 3 hours until a light green solution formed, indicating the formation of nickel hydroxide species. The resulting solution was stored in an airtight container for 24 hours to allow complete precipitation.

The precipitate was collected by centrifugation at 4000 rpm for 15 minutes and washed several times with distilled water to remove impurities. The cleaned precipitate was dried at 80 °C for 15 hours, followed by grinding into a fine powder using a mortar and pestle. Finally, the powder was annealed at 350 °C for 2 hours, producing pure NiO nanostructures with uniform morphology.

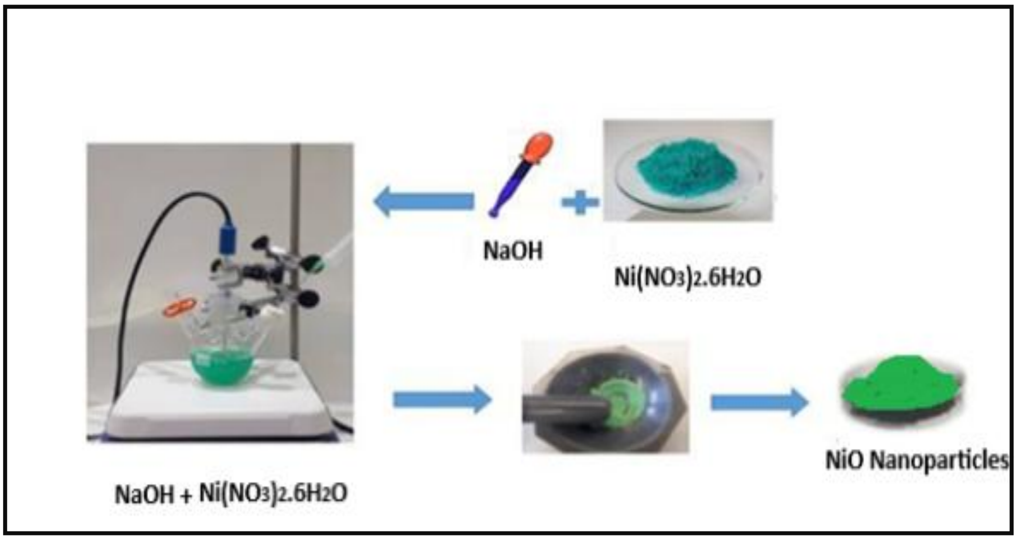

Fig .1

3. Characterization :

The NiO nanoparticle samples were investigated by using XRD and FTIR techniques. The Crystaline size of NiO nanoparticle were investigated by using the X-ray diffraction (XRD) method. FTIR spectra is obtained using a IR Affinity-1s from 400 to 4000 cm-2 using the KBr pellet technique and it used to identify chemical compounds.

1. RESULT AND DISSCUSSION A. X-Ray Diffraction (XRD):The purity and crystallinity of the as-synthesized NiO nanoparticles were examined by using powder X-ray diffraction (XRD) as shown in Figure1. It can be seen from Figure 2 that the diffraction peaks are low and broad due to the small size effect and incomplete inner structure of the particle. The peaks positions appearing at 2θ = 11.59◦, 34.12, 40.4◦, 59.70◦, and 71.64◦ can be readily indexed as (111), (200), (220), (311), and crystal planes of the bulk NiO, respectively. All these diffraction peaks can be perfectly indexed to the face-centered cubic (FCC) crystalline structure of NiO, not only in peak position, but also in their relative intensity of the characteristic peaks, which is in accordance with that of the standard spectrum . The XRD pattern shows that the samples are single phase and no any other impurities distinct diffraction peak except the characteristic peaks of FCC.

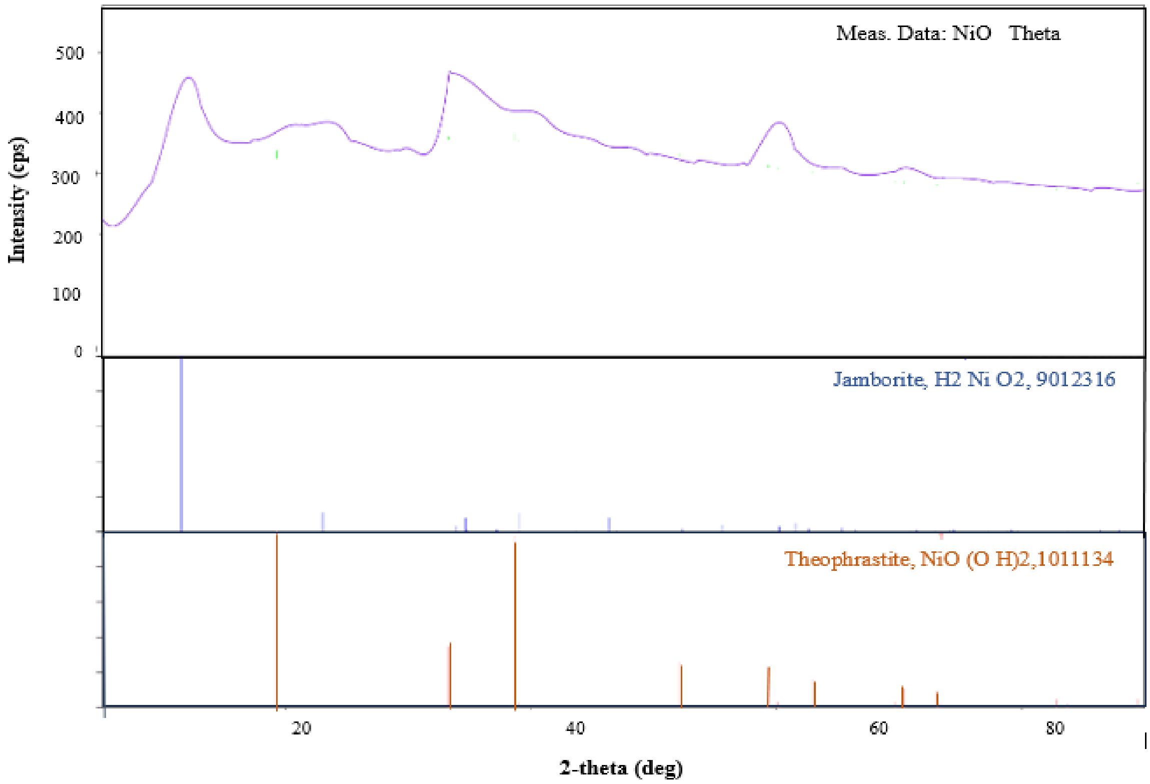

Fig .2

Phase NiO was detected. This result shows that the physical phases of the NiO nanoparticles have higher purity prepared in this work.[3]

The crystalline size of NiO NPs was found to be 18 nm which was calculated from measured values for the spacing of the (111) and (200) plane. which was estimated from the FWHM (Full Width Half Maxima) and peak position of an XRD pattern by Scherrer's formula.

$$D = \frac{0.94\lambda}{\beta cos\theta}$$

where D is the average crystalline size, λ is a wave length in Å, β is the FWHM in radian and θ is diffraction angle in degree.

2. Fourier Transformation Infrared Spectroscopy (FTIR) :

To understand the dominant functional group present in the NiO FTIR was recorded at room temperature. the FTIR spectra of NPs are as shown in Fig.3 From the FTIR spectra, peak around 447.50cm^{-1}, 644.25 cm^{-1} is because of the nickel oxygen bond stretching vibrations. The broadness of a peak indicates that the NiO catalyst is crystalline in nature.1054.14cm^{-1} is because of H-O-H bending. The peak1470.79cm^{-1},1629.92cm^{-1} indicates the existence of carbonate . 2418.84cm^{-1} , 2029.20cm^{-1} correspond to the C-H stretching mode .Also the broad peak 3446.94 cm^{-1} is because of stretching and bending vibrations of -OH group absorbed on catalyst surface from the atmosphere when FTIR analysis was carried out.

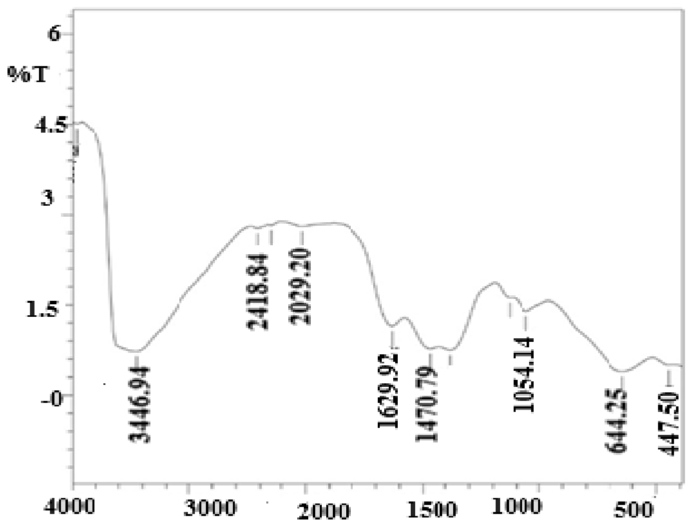

Fig.3

Conclusion :

The nanoparticles of Nio have been successfully synthesized by co-precipitation method. This method is simple, cost-effective, and easy to carry out. The process allows good control over particle size and produces Nio nanoparticles with enhanced surface area and improve physical and chemical properties. The analysis uses the (111) and (200) planes to estimate the size from the FWHM and peak positions. The conclusion typically highlights that the nanoparticles are nanocrystalline with a size of ~18 nm, suggesting good phase purity and small grain size, which can influence material properties like reactivity or optical behaviour. The ftir spectrum indicates the presence of O-H (hydroxyl), C-H (alkane), and C-O group.

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