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CO-PRECIPIATION SYNTHESIS AND CHARACTERIZATION OF NICKEL OXIDE (NiO) NANOPARTICLE

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Abstract

Nickel oxide (NiO) nanoparticles were successfully synthesized using the co-precipitation method. The resulting products were subjected to calcination at 400°C and subsequently analyzed using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and UV-vis absorption spectroscopy. The obtained findings confirm the presence of nickel oxide nano powders created during co-precipitation. The XRD patterns revealed the synthesis of cubic crystal structure NiO NPs, with a preferred orientation of (200). The FT-IR spectra exhibited a distinct absorption peak at approximately 550 cm⁻¹, indicating the presence of the Ni-O bond in the NiO NPs. Additionally, the UV-vis absorption spectra indicated that the NiO NPs had an absorption edge wavelength of 245 nm and an energy band gap of 4.5 eV.

Keywords: Nickel oxide, co-precipitation synthesis, X-ray diffraction studies, FT-IR spectra, UV-Vis spectra.

1. Introduction

In recent years, there has been significant interest in nanocrystalline transition metal oxides, particularly nickel oxide (NiO), due to their diverse potential applications. NiO is particularly appealing due to its chemical stability and exceptional performance in electrochemistry, optics, electronics, photoelectric catalysis, magnetism, thermal conductivity, mechanical strength, and chemical reactivity [1]. Nanoparticles exhibit unique properties distinct from those of bulk solid materials. These properties arise from various factors, including the small size effect, surface effect, quantum size effect, and macroscopic quantum tunnel effect [2, 3]. The one-dimensional structures of NiO, such as wires, rods, and ribbons, have garnered considerable attention for their ability to combine essential diagnostic, imaging, delivery, and dosage properties while remaining functionalizable. These materials offer significantly larger surface areas compared to bulk materials and can serve as both structural and functional components in devices [4]. Additionally, nanocrystalline NiO powder exhibits a super paramagnetic effect, making it suitable for applications in drug delivery and magnetic resonance

imaging (MRI) [5]. NiO is classified as a p-type (hole-type) semi-conductor with a wide energy band gap ranging from 3.6 to 4.0 eV, which can be influenced by cation vacancies. Various techniques, such as co-precipitation, hydrothermal methods, sol-gel techniques, and solvothermal methods, are available for synthesizing NiO nanoparticles [6-9]. During the synthesis process, numerous factors impact the properties of NiO nanoparticles, including the temperature of the precursor solution, the rate of alkaline solution addition, and the pH of the solution during nanoparticles growth [10]. A method for the preparation of NiO nanoparticles that is both feasible and facile is highly sought after. In this study, a co-precipitation technique is employed to synthesize NiO nanoparticles in a simple and efficient manner [11]. This method enables the mixing of particles at the molecular level and the processing of precursors and raw materials at low temperatures, resulting in the production of nanostructure powders and films.

2 Experimental

Materials used

Nickel nitrate hexahydrate [Ni (NO₃)₂·6H₂O] and sodium hydroxide [NaOH] were obtained from Merck, while deionized water was utilized for the solution preparation. The final product was washed using absolute ethanol.

Experimental method

In a typical experiment for synthesizing nickel oxide nanoparticles, a 0.25M aqueous solution of nickel nitrate hexahydrate and a 0.5M aqueous solution of sodium hydroxide were prepared separately in distilled water. The NaOH solution was heated to approximately 60°C, and the Ni (NO₃)₂·6H₂O solution was added drop wise to the heated solution while stirring at high speed for 2 hours. The resulting light green solution was left for 2 hours, and the light green precipitate was filtered and

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washed with deionized water and ethanol before being dried in a hot air oven at 80°C for 3 hours. The dried material was then calcined at 400°C for 4 hours, resulting in a black powder. Finally, the calcined powder was slightly crushed to obtain pure NiO nanoparticles. The reactions that occur during the experimental procedure are briefly summarized and reported in [12].

3 Results and Discussion

XRD analysis

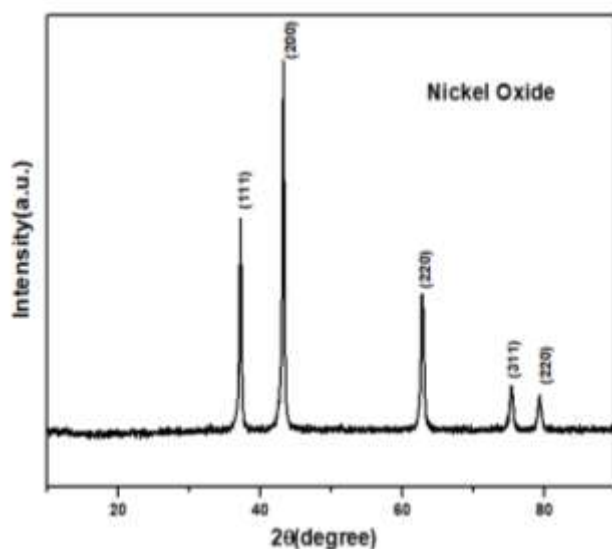


Figure 1: XRD pattern of NiO Nanoparticles

The XRD studies were conducted to characterize the phase purity of the sample. Figure 1 illustrates the XRD pattern of the NiO nanoparticles synthesized. The XRD peaks were observed at angles (2θ) of 37.26°, 43.50°, 63.08°, 75.39°, and 79.50°, corresponding to the (111), (200), (220), (311), and (222) planes, respectively. These peaks demonstrate a strong agreement with the cubic crystal structure of NiO (JCPDS Card No: 78-0643; space group = Fm3/m). The grain size of the crystallites in the synthesized product was determined using the Debye-Scherrer formula

$$d = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

The nanoparticles synthesized had an average size of 21 nm. In this case, λ denotes the wavelength of the X-ray used (0.15418 nm), β represents the full width at half-maximum of the radiation peak, and θ represents the Bragg angle of the X-ray diffraction peak.

FT-IR Spectra

Figure 2 presents the FTIR spectrum of NiO nanoparticles. The absorption band at 3425 cm^{-1} is attributed to OH stretching mode, while the weak peak at 1627 cm^{-1} corresponds to OH bending mode of water [13]. The wide absorption band around 1381 cm^{-1} can be attributed to CO_3^{2-} ions. The peak at 1033 cm^{-1} corresponds to the stretching and bending vibrations of the intercalated C-O species [14], and the bands at 2924 cm^{-1} and 2854 cm^{-1} can be assigned to CH_2 vibrations.

Additionally, the observed peak at 550 cm^{-1} corresponds to the stretching mode of Ni-O nanoparticles [15].

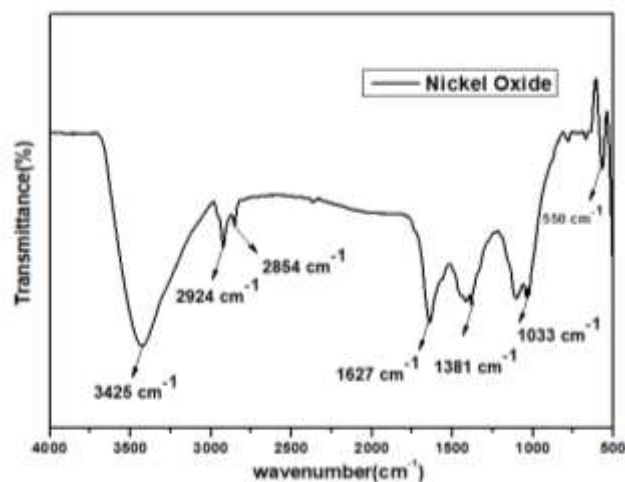


Figure 2: FTIR spectrum of NiO nanoparticles

UV-Visible absorption spectra

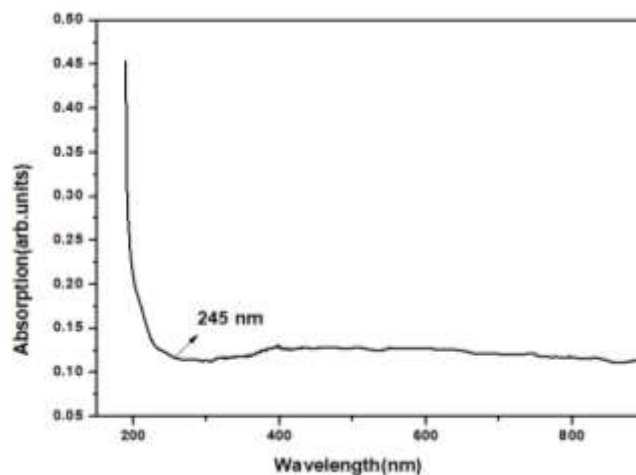


Figure 3 (a): UV-Visible Spectrum of NiO Nanoparticles

Figure 3(a) illustrates the UV-visible spectrum of the NiO nanoparticles suspension achieved through ultrasonic dispersion in water. A prominent absorption peak in the UV region is observed at a wavelength of 272 nm. This absorption in the UV region is attributed to the band gap absorption of NiO [16]. It is predicted that the top of the valence band consists of the oxygen 2p band, while the bottom of the conduction band is primarily derived from Ni 3d states [17].

Figure 3(b) presents the band gap spectrum of NiO nanoparticles. The energy band gap of NiO nanoparticles was determined using the well-known Tauc relation [18]

$$(Ah\nu)^n = A (h\nu - E_g)$$

Here, α represents the absorption coefficient, $h\nu$ denotes photon energy, A signifies absorbance, E_g represents the optical band gap, and n characterizes the nature of the transition process; $n = 2$ for direct transition and $n = 1/2$ for indirect transition. Consequently, the optical band gap for the absorption edge can be obtained by extrapolating

the linear portion of the $(\alpha h\nu)^2$ vs $h\nu$ plot. The calculated band gap energy from this plot is found to be 4.5 eV.

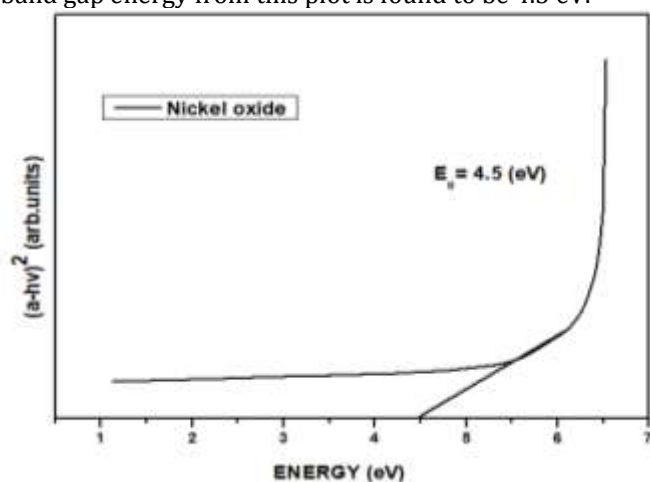


Figure 3 (b): Band Spectrum of NiO Nanoparticles

4 Conclusions

NiO nanoparticles were synthesized using co-precipitation method utilizing nickel nitrate hexahydrate and sodium hydroxide. XRD analysis confirmed the nanocrystalline structure of the particles, with a crystallite size of 21 nm at a calcinations temperature of 400°C. FTIR spectrum indicated the formation of NiO nanoparticles, while diffuse reflection spectroscopy revealed an optical band gap of 4.5 eV and an absorption band edge of 245 nm.

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