

Synthesis, Characterization, and Magnetic Properties of Pure and Mn doped NiO Nano Particles

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Abstract:

The influence of Nickel(II) acetate tetrahydrate as a capping agent on the structure, morphology, optical properties, and magnetic properties of nickel oxide (NiO) nanosized particles, which were synthesized using the co-precipitation method, was investigated. Nickel chloride hexahydrate and sodium hydroxide (NaOH) were utilized as precursors in the synthesis process. The resulting nanoparticles were thoroughly examined using X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). The XRD patterns revealed that NiO possesses a face-centered cubic (FCC) structure. The size of the crystallites, as estimated by the Scherrer formula, ranged from 28 to 33 nm. Notably, the NiO nanoparticles exhibited a smaller size compared to the pure nanoparticles. This suggests that the addition of the capping agent, nickel acetate tetrahydrate at a concentration of 0.1M, facilitated the formation of nucleation points for nanoparticle growth. The optical and magnetic properties of the nanoparticles were studied using Fourier Transform Infrared Spectroscopy (FTIR), UV-vis Absorption Spectroscopy (UV), Vibrating Scanning Magnetometer, and magnetization measurements. The FTIR spectra indicated the presence of absorption bands in the range of 400-4000 cm-1, which is a characteristic feature of NiO. The magnetic hysteresis of the NiO nanoparticles demonstrated that the capping agent enhanced the surface magnetization of the nanoparticles. Furthermore, cyclic voltammetry (CV) and impedance spectroscopy were employed to evaluate the electrochemical performance of the nanoparticles in a 1M NaOH solution. The NiO modified electrode exhibited higher electroactivity and faster electron transport at the electrode surface compared to the bare electrode and Mn/NiO electrodes.

Keywords: Nickel Oxide, Elemental quantitative information, X-ray Diffraction, Cyclic Voltammtry, and Vibrating Scanning Magnetometer

Introduction

Nanoscale nickel oxide (NiO) is a significant transition metal oxide that has gained attention as a promising candidate in various fields, including superparamagnetic devices, photovoltaic devices, electrochemical supercapacitors, magnetic materials, catalysis, smart windows, fuel cells, and photovoltaic devices [1]. These nanostructured particles are considered as a p-type semiconductor with a large exciton binding energy and a stable wide bandgap of 3.6–4.0 eV. In its bulk form, NiO is an antiferromagnetic insulator with a Néel temperature of 523K[2]. Due to their quantum size and surface effects, these nanoparticles exhibit unique magnetic, optical, electronic, and chemical properties that differ significantly from those of bulk-sized NiO particles [1].

Several methods have been utilized to prepare NiO nanoparticles, such as the sol-gel process [3], thermal decomposition [4], polymer-matrix assisted synthesis [5], and spray-pyrolysis [6]. However, some of these techniques face challenges in achieving size homogeneity and dispersion of NiO nanoparticles. Generally, most techniques aim to reduce the costs of chemical synthesis and produce materials suitable for technological applications [7]. In solution-based chemical methods for nanoparticle synthesis, a capping agent is typically



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added to control the size and shape of the nanoparticles and prevent their agglomeration. Various organic compounds, such as surfactants and polymers, have been used to direct or confine anisotropic growth. Among the different methods for controlled synthesis, we employed a Co-Precipitation chemical route based on the solution process to synthesize NiO nanoparticles. Using surfactant-mediated synthesis, we prepared NiO and Mn nanocrystalline particles with an average diameter of 28-33 nm. However, the NiO nanoparticles did not disperse well [8]. In another approach, we successfully prepared well-dispersed NiO nanoparticles with an average particle diameter of 30 nm using polyvinylpyrrolidone as an assisting agent. These nanoparticles find wide applications in various fields, leading to the formation of cobalt with different substituents, resulting in different structural motifs. Recent studies have shown increased interest in cobalt complexes with substituted organic groups such as isothiocyanates [9], dithiolanes [10], cyanamides [11], heterocyclic rings [12], and hydroxyl groups [13]. Even slight variations in these attached groups significantly affect the complex, resulting in an interesting array of structures [14]. Manganese compounds possess an interesting property of volatility, making them suitable precursor compounds for metal oxides [15].

To explore the electrocatalytic and charge transfer properties of the synthesized nanoparticles, cyclic voltammetry and impedance spectroscopy are commonly employed electrochemical techniques. The modification of electrodes with nanoparticles has been reported to improve their properties compared to unmodified electrodes. This study is crucial in confirming the practical application of the synthesized nanoparticles, especially in the field of electrochemical sensors. In this paper, we synthesized single-phase Heazlewoodite nickel acetate and Bunsenite nickel oxide through the Co-Precipitation method using hexadecylamine and calcination, respectively. We present the optical properties and morphology of the nanoparticles, along with the utilization of electrochemical techniques to study the redox behavior of the synthesized nanoparticles.



Experimental Procedure

Flowchart of Pure NiO and Mn doped NiO Nanoparticles.

In this study, I have chosen to investigate the synthesis of pure and Manganese doped NiO nanoparticles using the co-perception method. All the chemical reagents used in our experiments were of high quality and obtained from the sigma-Aldrich company. We did not perform any additional purification on these reagents. To synthesize the nanoparticles, we first dissolved Nickel(II) acetate tetrahydrate [(CH3COO)2NiO.4H2O] in 50ml



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of double distilled water. Then, we weighed and dissolved the samples with a stoichiometry of NiO 0.99 Mn 0.01 in de-ionized water. The solutions prepared from the starting materials were placed in a round-bottomed flask with a reflex cooling tube. We gradually added an aqueous solution of 5mol/l NaOH to the stoichiometric solutions and stirred them magnetically for 30 minutes at room temperature. Afterward, the precipitate was obtained and allowed to settle for 3 hours. The resulting green precipitate was filtered out. The stirring process was continued at room temperature until the pH approached 13. At this point, we added another dopant, MnCl2.4H2O, in the range of 0.01mol% to 100ml solution. This mixture was added to the pre-prepared mixture and stirred magnetically for 1 hour at room temperature. Then, dropwise addition of an aqueous solution of 0.1 M NaOH was done to the above mixture at room temperature, and the final precipitate was obtained. It was then centrifuged and washed several times with double distilled water, followed by ethanol, to remove any watersoluble byproducts (peptization).

The resulting precipitates were filtered using fine pore size filter paper, and the spongy content in the filter cone was allowed to dry. The collected colloidal precipitate was dried in a micro hot air oven at 120°C for 24 hours. The dried product was subsequently annealed at 550°C for 12 hours. After annealing, the dried powder samples were ground in an agate mortar. The samples prepared using pure and Mn doped nanoparticles were labeled as N pure and doped NiO1, and pure and Mn doped NiO2, respectively. To analyze the structure and phase of the prepared samples, we conducted X-ray diffraction studies using an X-ray diffractometer (XPERT-PROPW050) at room temperature. The surface morphology was examined using a scanning electron microscope (SEM Zeis super 55VP). The UV-Visible absorbance spectra were recorded using a spectra photos (JASCOV-570) in the range of 200nm-800nm to determine the band gap of the prepared samples. Additionally, particle size analysis was performed using dynamic light scattering (DLS). Finally, we carried out magnetization measurements (M-H Characterization) of the samples using a vibrating sample magnetometer (Lake share model:74047).

Results and discussion

3.1 X-Ray Analysis:

The X-ray diffraction (XRD) patterns of the Pure NiO and Mn-doped NiO nanoparticles exhibit distinct peaks that are absorbed at specific 2θ values, as shown in Figure 1. To determine the average size of the nanoparticles, the Scherer formula is employed, which is expressed as

$$Dh,k,l=(0.9\lambda/\beta h,k,l\cos\theta) \dots (1)$$

where λ represents the wavelength ($\lambda = 1.542$ Å) (CuK α), β denotes the full width at half maximum (FWHM) of the line, and θ represents the diffraction angle. By analyzing the XRD patterns, the size of the Pure NiO nanostructure is found to be approximately 28 nm when examining the reflection from the (200) plane, as indicated in Figure 1. It is evident from the figure that all the samples exhibit identical diffraction peaks, namely (200), (220), (311), (400), and (420), which correspond to the face-centered cubic structure of Mn-doped NiO nanoparticles. These diffraction peaks align with those observed in the standard spectrum (JCPDS, No.01-073-1523) for the X=0 sample. However, due to the nanoscale size of the material, the peaks at higher angles are obscured by the background, resulting in broadening of the lines. Consequently, the average size of the NiO nanoparticles, as indexed to the (200) plane in the XRD pattern, is determined to be 28 nm.







3.2 UV – Analysis

The confirmation of the creation of Nickel Oxide nanoparticles doped with Pure and Mn has been established through the detection of Surface Plasmon absorption peaks in the UV-VIS spectra of the samples, which were prepared using the co-precipitation method. It is notable that the wavelengths corresponding to the peak absorption exhibit a blue shift in their spectrum. The presence of an absorption peak ranging from 200 to 800 nm further supports the formation of Pure and Mn doped NiO nanoparticles. Interestingly, the absorption initially decreases significantly during progressive scanning, with the estimated absorption coefficient (α) approaching zero without notable variations up to the scanning limit of 800 nm[16]. The observed spectra (Fig. 2) reveal an absorption occurrence at the wavelength edge of 331 nm for both pure NiO and Mn doped NiO samples.



Fig.2. UV-vis Spectrum of Pure NiO and Mn doped NiO nanoparticles

3.3 FTIR Analysis

The identification and prediction of the functional groups present in Pure and Mn-doped NiO nanoparticles were accomplished using FT-IR spectroscopy. The spectroscopic analysis revealed distinct absorption bands at 1415, 1615, and 1112 cm-1, as depicted in figure 3. The peak observed at 1415 cm-1 signifies the predominant presence of O-H, which acts as a reducing agent. Furthermore, the peak at 1615 cm-1 indicates the stretching vibrations of the C-H bonds in the –CH2 functional group [17]. Additionally, the peaks at 998 cm-1 and 613 cm-1 correspond to the bending of aromatic compounds and the asymmetric vibration of sulfate groups [18]. Lastly, the peak at 1615 cm-1 represents the stretching band of the C-O bonds. In the spectra of pure NiO and Mn-doped NiO nanoparticles displayed in figure 3, all these bands remain visible. However, specific to the spectra of these nanoparticle samples, new peaks appear at 613 cm-1, indicating the presence of the aromatic C-H bending band. Peaks at 577 cm-1 and 515 cm-1 further confirm the successful synthesis of pure NiO and Mn-doped NiO nanoparticles, respectively, as they correspond to the Ni-O stretching vibration modes. The presence of the metal oxygen band at 577 cm-1 suggests the stretching vibration of the metal at the tetrahedral site, while the peak at 515 cm-1 indicates the presence of the metal at the tetrahedral site, while the peak at 515 cm-1 indicates the presence of the metal at the octahedral site [19].





3.4. Scanning electron microscopic (SEM) & energy dispersive X-ray (EDX) analysis

The SEM technique was utilized to examine the morphology of the as-grown nanostructures. The SEM images in Figure (4.a-d) depict the synthesized product. It is evident that in this particular case, successful preparation of Pure NiO and Mn doped NPs were achieved. Scanning electron microscopy measurements were employed to determine the particle size and morphology of the synthesized materials. The SEM photographs of NiO material calcinated at different doped levels are displayed in Figure (4 a-d). Notably, there are distinct differences in particle size depending on the calcination temperature. The particle size ranges from 25 to 80 nm. When comparing the materials calcinated at 150°C, it is observed that pure NiO exhibits smaller particle size compared to Mn doped materials. The reason for these differences in particle size is likely due to an enhanced particle growth rate at higher calcination temperatures. The slightly spherical-like shape utilized in this study allows for a higher combination of reactant molecules and more active areas, as demonstrated in Figure (4.a-b) where pure NiO exhibits a spherical-like morphology nanostructure. From Figure (4.c-d), it can be seen that Mn doped NiO indicates the formation of nanoparticles. To further analyze the pure NiO and Mn doped NiO samples, energy dispersive X-ray analysis (EDX) was conducted, as shown in Figure (4 e and f). EDX analysis was performed on NiO nanoparticles at 10 keV. The analysis revealed the presence of Nickel (Ni) and oxygen (O) elements in NiO NPs, indicating that the nanoparticles were almost stoichiometric. The mass percentages of nickel and oxygen determined from the EDX examination were 95.43 wt% (0.804 keV) and 3.57 wt% (0.804 keV), respectively, with no additional elemental impurities detected in the pure EDX spectrum. From Figure (4.f), the Mn doped NiO NPs displayed the occurrence of Nickel (Ni), oxygen (O), and Manganese (Mn) elements. The weight percentages calculated from the EDX analysis for Mn doped NiO were 1.1 wt% (0.4 keV) for Mn. This finding confirms the successful synthesis of pure NiO nanoparticles.





Fig.4. (a-b) SEM image of pure NiO and; (c-d) Mn doped NiO nanoparticles; (e) EDAX spectrum of pure NiO and; (f) Mn doped NiO nanoparticles.

3.5. Vibrating Sample Magnetometer (VSM) Observation

Magnetization measurements were conducted on NiO nanoparticles using the VSM technique within a range of -15000 to 15000 Oersted at room temperature. The results, depicted in Figure 5, illustrate the nanoparticle's magnetization before and after the doping process. The VSMs utilized in this study operated at room temperature with an applied magnetic field of 10 kOe. These findings provide valuable insights into the behavior of saturation magnetization, coercive and residual magnetization, and M-H hysteresis loops in the context of NiO nanomaterials. Notably, the NiO nanoparticles demonstrate superparamagnetic properties at room temperature. The VSM results exhibit a distinct S-shaped curve in response to the applied magnetic field. Incorporating additives into the NiO nanostructure leads to a decrease in both the saturation magnetizations decrease with increasing Mn concentration in the NiO nanomaterial. This phenomenon can be attributed to the strain experienced at the grid point, as previously reported in other studies [20, 21]. The coercive effect of pure NiO and NiO with incorporated additives is greatly influenced by factors such as anisotropy, particle morphology, porosity, micro-expansion, size distribution, and the magnetic domain's size, which can all be characterized and measured. It has been observed that Mn, being an element with inherently soft magnetic characteristics, may lose some of its soft nature upon incorporation with NiO.

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Fig.5.VSM Analyser of Pure and Mn doped NiO nanoparticles

3.6. Electrochemical characterization

The cobalt electrode was enhanced by utilizing a synthesized nickel oxide nanoparticle in order to investigate their electrochemical properties. The presence of a nanoparticle layer on the electrode was verified through cyclic voltammetry and impedance spectroscopy conducted in a 1 M NaOH solution. The electrode was subjected to a potential range of -1.5V to 2.0V with a sweep rate of 20 mVs-1. It is evident that there are distinct redox peaks, indicating that the capacitance characteristics are influenced by Faradic reactions [22]. The occurrence of these redox peaks is observed in both samples, attributed to the transition of Ni2+ to Ni 3+ at the surface of NiO NPs as described by the following equation [23, 24].



Fig.6. Comparative cyclic voltammograms of Pure and Mn doped NiO nanoparticles

Fig. 6 illustrates a comparative analysis of cyclic voltammograms of NiO and Mn/NiO modified electrodes in a 1 M NaOH solution at a scan rate of 20 mVs-1. The electrodes modified with nickel oxides (NiO) exhibited

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superior voltammograms compared to those modified with manganese/nickel oxides (Mn/NiO). However, the modified electrodes demonstrated no significant response, as depicted in Fig. 6. This lack of response could be attributed to the presence of hexadecylamine acting as a capping agent for the NiO nanoparticles. While this capping agent positively hindered agglomeration, it also impeded the interaction between the nanoparticles, the surface of the Mn electrode, and the electrolyte. Consequently, this hindered interaction affected the electrochemical behavior of the modified electrodes [25].

Fig. 6 (a) and (b) display the cyclic voltagrams of NiO. In the initial cathodic sweep, a prominent peak was observed at 0.39 V, corresponding to the first electrochemical process of NiO. This process led to the formation of Ni-metal nanoparticles, amorphous Mn, and the solid electrolyte interface (SEI) layer. The SEI layer is comprised of ethylene oxide based oligomers, LiF, LiCO3, and lithium alkyl carbonate [26, 27]. Subsequent cycles showed a weaker peak at 1.12 V, indicating the reversibility of the electrochemical reactions. In the anodic sweep, two broad peaks at 1.5V and 2.28V were observed, which can be attributed to the decomposition of the SEI layer and the oxidation of Ni to NiO, respectively. Finally, this decomposition process also involves Mn.



Fig.7. Typical Nyquist plots obtained for Pure and Mn doped NiO nanoparticles

Electrochemical impedance spectroscopy (EIS) is a highly effective technique employed to examine the electron transfer characteristics of electrodes that have been altered on their surface. The graphical representations, known as Nyquist plots, depicting the findings for the modified electrodes can be observed in Figure 7. The augmentation in both Z components is attributed to a modification in the resistance for electron transfer, which is caused by the presence of a biocomposite layer on the electrode's surface. This biocomposite layer also leads to a reduction in capacitance due to the increased separation distance between the electrode surface and the electrolyte solution. Notably, significant alterations in impedance components are observed solely at lower frequencies. Additionally, the Nyquist plots for the high and medium frequency regions are also displayed in Figure 7. The peak frequency for the imaginary part is determined for both the unmodified and modified electrodes. These outcomes indicate that NiO and Mn/NiO possess exceptional electrochemical properties, such as heightened sensitivity, reduced charge transfer resistance, stability, and well-defined redox voltammogram. Consequently, it is evident that the proposed modified electrodes hold potential for utilization in electrochemical sensor applications.

Conclusion:

To summarize, we have successfully synthesized Pure NiO and Mn-doped NiO nanoparticles using a simple Co-Precipitation method at room temperature. The XRD patterns of the nanoparticles indicate a face-centered cubic

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structure for the Mn-doped NiO nanoparticles. The UV-Vis absorption spectra show a blue shift in the peak wavelengths. We used FT-IR spectroscopy to determine and predict the functional groups present in both the Pure and doped nanoparticles. In this study, the spherical-like shape of the nanoparticles enabled a more efficient combination of reactant molecules, as observed in the SEM images. We analyzed the resultant solution of the samples using energy dispersive X-ray analysis, which confirmed the presence of pure and Mn-doped NiO nanoparticles. Remarkably, the nanoparticles exhibited superparamagnetic behavior at room temperature. The VSM results showed a well-defined S-shaped curve, indicating the sample's response to the applied magnetic field. The CV profiles of NiO revealed an intense peak at 0.39 V during the first cathodic sweep, corresponding to the first electrochemical process of NiO. This process resulted in the formation of Ni-metal nanoparticles, amorphous Mn, and a solid electrolyte interface (SEI) layer. The Nyquist plots obtained for the modified electrodes demonstrated an increase in both Z components, which can be attributed to a change in the electron-transfer resistance caused by the biocomposite layer on the electrode's surface. This change also led to a decrease in capacitance due to the increased distance between the electrode's surface and the electrolyte solution.

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