Synthesis and Characterization of NiO Nanoparticles by Thermal Decomposition Method

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Abstract—Nickel oxide (NiO) nanoparticles were prepared by Thermal Decomposition method. Thermal decomposition or thermolysis is a chemical decomposition caused by heat. Thermal decomposition was thoroughly studied in order to control the particles size of the as-prepared NiO Nano powders. Transition metal oxides and metals have been researched extensively due to their interesting catalytic, electronic and magnetic properties. It has more applications like electro-chromic coatings, plastics, textiles, nanowires, Nano fibers and specific alloy and catalyst applications. It is also used in active optical filters, ceramic structure, p-type transparent conductive films and energy efficient smart windows. The synthesized nickel oxide (NiO) nanoparticles were subjects to various studies like X-ray diffraction (XRD) technique and Fourier Transfer Infrared spectroscopy (FTIR).

Key words: Nickel Oxide, Thermal Decomposition Method, XRD, FT-IR

I. INTRODUCTION

Transition metal oxides and metals have been researched extensively due to their interesting catalytic and electronic properties. Nickel oxide (NiO) has a wide band gap [1] and varied applications [2]. They include catalysis[3], lithium ion batteries[4,5], smart windows[6], anti-ferromagnetic film[7], dye-sensitized photocathodes[8,9], photo catalysis[10], anti-microbial activity[11], thermal conductivity[12] and field emission studies[13]. Varying the particle size of NiO leads to novel and interesting magnetic properties that range from nano sized to bulk –like behavior [14-30]. Nanoparticles have been prepared by different methods such as solvo thermal process [31], Thermal decomposition [32, 33], sol-gel [34] and reverse micro-emulsion method[35]. In the present study, metal oxide Nanophases such as Nickel oxide have been synthesized by the method of thermal decomposition using nickel oxalate dehydrates. The products were characterized by powder X-ray diffraction and Fourier Transform Infrared Spectroscopy (FTIR).

II. EXPERIMENTAL PART

A. Materials:
- Nickel oxalate ( AR grade MERCK)

B. Synthesis:
-Synthesis of Nickel Oxide

A small amount of the nickel oxalate has been used with alumina crucible for thermal treatment. Isothermal treatments have been carried with a furnace at a temperature of 450° C for two hours respectively. The temperature has been recorded with an accuracy of better than 1° C. The heating rate has been maintained at 10K/min. A structured

III. RESULTS AND DISCUSSION

A. Structural Characterization:

1) XRD:
The X-ray diffraction pattern of the synthesized Nickel oxide nanoparticles is shown in fig.1 & 2 and the peak details are in Table.1. The experimental XRD pattern agrees with the JCPDS card no.89-7390 and the XRD pattern of NiO nanoparticles other literature. The 2θ at peak confirms the NiO. Strong diffraction peaks at 37° and 43° indicating the NiO. The intensity of XRD peaks of the sample reflects that the formed nanoparticles are crystalline and broad diffraction peaks indicate very small size crystallite.
IV. PARTICLE SIZE CALCULATION

From this study, considering the peak at degrees, average particle size has been estimated by using Debye-Scherer formula. The results show that the value for the average particle size is 46nm for the nickel oxide specimen. Interplanar spacing between atoms (d-spacing) is calculated using Bragg’s law and enumerated in Table.1.

\[ D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1) \]
\[ 2d \sin\theta = n\lambda \quad (2) \]

The values of particle size have also been computed by using the Hall-Williamson method. Subsequently, plot of \( \beta \cos\theta \) as a function of \( \sin\theta \) as per equation (3) for the nickel oxide specimen is shown in fig 3. The results show the value of particle size obtained by this method is 71.84nm which is higher than those obtained by the Scherrer method as from equation 3.

\[ \beta \cos\theta = \left(0.9/\lambda\right) + \eta \sin\theta \quad (3) \]

Where, \( \lambda \) is wavelength of X-Ray (0.1540nm), \( \beta \) is FWHM (full width at half maximum), \( \theta \) is diffraction angle, \( d \) is d-spacing and \( D \) is particle diameter size and \( n \) is the order of diffraction, \( \eta \) is a function of lattice strain in the specimen.

V. DISLOCATION DENSITY, MACROSTRAIN AND MICROSTRAIN

A dislocation is an imperfection in a crystal associated with the misregistry of the lattice existing in different parts of the crystal. The dislocation density can be calculated using the formula 4.

\[ \rho = \frac{1}{T^2} \quad (4) \]

The micro strain can be calculated using the formula 5, and the macrostrain can be calculated using the formula 6. The values of dislocation density, macrostrain and micro strain are shown in the table 2.

\[ \epsilon = \beta \cos\theta/4 \quad (5) \]
\[ \epsilon = \frac{d_{\text{obs}} - d_{\text{std}}}{d_{\text{std}}} \quad (6) \]

where \( \rho \) is the dislocation density of the material, \( \epsilon \) is the macrostrain and \( d \) & \( d \) are the observed and standard interplaner spacing for different crystal planes with different miller indices(hkl).

<table>
<thead>
<tr>
<th>S.N. o.</th>
<th>Transmittance bands (cm(^{-1}))</th>
<th>Band assignment</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>435.93 cm(^{-1}) 430 cm(^{-1})</td>
<td>Stretching Vibrion of Ni-O</td>
</tr>
</tbody>
</table>

Table 3: FTIR spectroscopy data for the Nickel Oxide specimen along with the band assignments and a comparison with literature

VI. FT-IR SPECTRAL ANALYSIS

Fig 4. Shows the FTIR spectra for the nickel oxide specimen. The spectrum has one profile 425 cm\(^{-1}\) due to stretching mode of vibrations of Ni-O bonds which confirms decomposition of the as-received specimen. The observed bands in the spectra are also presented in table 3.

![Fig. 4: FTIR spectra for the nickel oxide specimen](image-url)
VII. Conclusion

NiO nanoparticles were prepared by using thermal decomposition method. The synthesized nanoparticles were characterized by XRD, FTIR spectroscopy. The XRD pattern showed that the synthesized NiO nanoparticles were crystalline in nature and they have Rhombohedral structure and the average particle size is 46nm. The FTIR study confirmed the functional groups appeared at 425 cm$^{-1}$ in NiO nanoparticles were due to the stretching of NiO.

References