

Effect Of Different Temperatures On Structural And Magnetic Properties Of Nickel Oxide Nanoparticles By Microwave Assisted Solvo Thermal Method

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Abstract : Nanostructured electrode materials exhibit more attractive properties compared to electrodes materials. NiO has been considered as one of the promising potential electrode materials due to its large surface area, high conductivity, environmental friendliness and its low cost. In this study NiO nanoparticles have been synthesized by microwave assisted solvothermal method. Analytical grade Nickel acetate, Urea and Ethylene Glycol are used as precursors. The prepared sample was annealed at 300°C, 400°C & 500 °C for 1 hr. The structural properties were investigated through X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. XRD result shows that the annealed NiO sample exhibited cubic crystalline structure with crystallite size of 7nm, 11nm & 20 nm. The functional group analysis of the sample was studied by Fourier Transform Infrared spectroscopy (FTIR). The magnetic behavior of the samples was studied by Vibrating Sample Magnetometer (VSM) and the results show that the NiO samples are ferromagnetic in nature at room temperature.

Keywords: Nanostructure nickel oxide, XRD, FTIR and VSM

1. INTRODUCTION

Research on new materials technology is attracting the attention of researchers all over the world. Developments are being made to improve the properties of the materials and to find alternative precursors that can give desirable properties on the materials. It has a wide range of application in chemistry, materials, sensors, electronics, information storage, communication, protection measurements of the environment, aerospace, biological systems, drug delivery systems, etc. NiO is an anti-ferromagnetic oxide semiconductor with p-type conductivity due to its wide band gap energy range from 3.6 to 4.0 eV [1]. The particle size has strong influence on the magnetic properties of NiO nanoparticles. Smaller particles have large interface area between the ferromagnetic phase and anti-ferromagnetic matrix, and the structural disorder and exchange coupling increases with interface area. Recently, several methods have been developed to prepare nickel oxide nano material powder including low pressure spray pyrolysis [2], sol gel [3], magnetron sputtering [4,5], thermal decomposition [6] and other techniques [7-9]. NiO nanoparticles with a uniform size and well dispersion are desired for many applications. Nickel oxide (NiO) nanocrystalline has been reported to have large surface areas, effective adsorptive properties and fast diffusion property [10]. Various types of metal/metal oxide nanoparticles have been extensively synthesized by chemical and mechanochemical methods which are widely used in catalysis, battery cathodes, gas and glucose sensors [11]. Here, the objective of the study is to find out the effects of NiO nanoparticles because change in different annealing temperature has exhibited drastic and interesting change in the structural and magnetic properties. In this work the Nickel oxide nanoparticles are synthesized using microwave assisted solvothermal method. Microwave heating has unique effects compared with the conventional heating such as rapid volumetric heating, selective heating and energy saving considerations. These advantages of microwave heating are

increased reaction rates, shorter reaction time and enhanced reaction selectivity. The different calcination temperatures of NiO nanoparticles is found by synthesis method, structural, and magnetic properties .

2. EXPERIMENTAL PROCEDURE

The precursors used for the synthesis of NiO nanoparticle were analytical grade Nickel acetate, urea as a catalyst and ethylene glycol as a solvent. Initially at room temperature, Nickel acetate and urea were dissolved in ethyleneglycol by constant stirring for 90 min in a magnetic stirrer. The microwave power was set to 650 watt and operated at the rate of 2 min per cycle and cooled in intervals until the precipitate was formed. The resulting precipitate was washed with double distilled water and dried. Again, the dried powders were washed with acetone and annealed at 300°C, 400°C & 500 °C for 1 hour in air atmosphere.

3. RESULTS AND DISCUSSION

3.1 THERMAL ANALYSIS

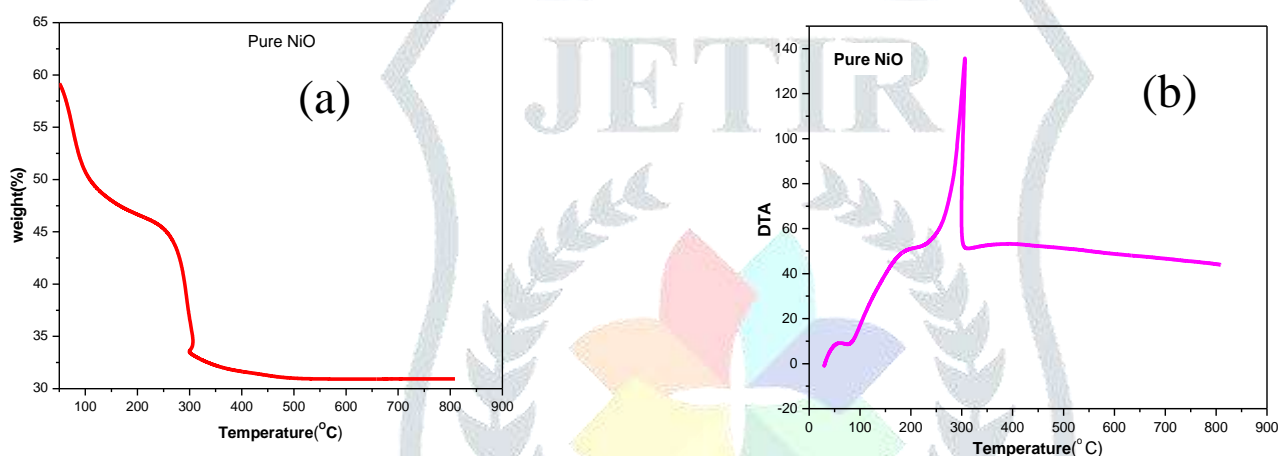


Fig. 1-(a) TGA and (b) DTA for pure NiO nanoparticles

The thermal observation of the prepared precursor was investigated with thermal gravimetric analysis (TGA) to find the suitable calcinations of the NiO nanoparticles (Fig.1a). In the present investigation, the weight of the nonstoichiometric oxygen is obtained directly by its weight loss. The thermo-gravimetric (TG) curve and its differential curve (DTG) are shown in Fig.1 (a) & (b) for NiO. It is clear from Fig.1b that a very sharp peak is present between 200°C and 300°C on the DTA curve, which is a measure of the amount of excess oxygen released in the heating process. Below 200°C, weight loss occurred because of the evaporation of absorbed water. In the temperature range of 200°C to 300°C, weight loss is due to the decomposition of precursor materials. This value suggested that the precursor decomposed completely at 300°C to become nickel oxide. Above 300°C, the constant weight loss occurred because the decomposition reaction was completed. When the powder was annealed at a temperature of 300°C or higher, there was almost no water according to the TG analysis. The powder was expected to be pure NiO. Therefore, only NiO powders annealed at 300 °C or higher were used in our study.

3.2 STRUCTURAL ANALYSIS

3.2.1. X-Ray Diffraction Patterns.

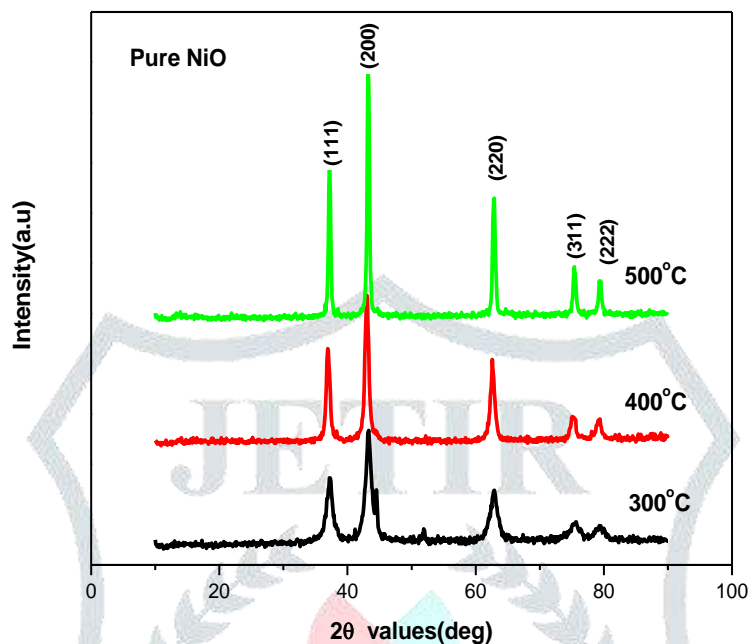


Fig. 2 XRD pattern of NiO Nanoparticles annealed at 300°C, 400°C and 500°C for 1 hr

Figure 1 displays the X ray diffraction patterns for pure NiO nanoparticles annealed at 300°C, 400°C & 500°C. The five dominant diffraction peaks at approximately $2\theta = 37.17^\circ$, 43.21° , 62.79° , 75.34° and 79.34° can be perfectly related to (111), (200), (220), (311), and (222) crystal planes, respectively. The sharpness and the intensity of the peaks are quite an indication of the well crystalline nature of the prepared nanoparticles. All these diffraction peaks can be perfectly indexed to the face centered cubic (FCC) crystalline structure of NiO which is in accordance with that of the standard spectrum (JCPDS NO: 78-0429). Similar patterns were observed by Gondal et al. for NiO particles prepared by pulsed laser ablation technique [12]. The crystallite size diameter (D) of the NiO nanoparticles has been calculated by Debye-Scherrer equation.

$$D = \frac{k\lambda}{\beta \cos\theta} \text{ metre}$$

where $\lambda = 1.54056$ nm is the wavelength of the X-ray, $\beta = \text{FWHM}$ (full width at half maximum), θ is diffraction angle obtained from 2θ values corresponding to maximum intensity peak in XRD pattern (200), and k is an empirical constant equal to 0.9. The diameters of the crystallite size calculated by the Scherrer formula are 7nm, 12nm & 20nm respectively. No other peaks of impurities were detected.

3.2.2 SEM AND EDAX ANALYSIS

SEM and EDAX techniques were also applied to characterize the synthesized nickel nanoparticles. SEM image of NiO nanoparticles is shown in Fig. 3a. From this image, it is clear that the diameters of the nanoparticles are close in agreement with the size estimated by XRD data. Each nanoparticle is mostly irregular spherical in shape. Some particles are found to be agglomerated. It indicates that the nanoparticles are homogeneous in size and well dispersed. The EDX spectrum is presented in Fig. 3b. It shows the peaks reflecting the presence of nickel and oxygen. The quantitative analysis is tabulated in Table 1.

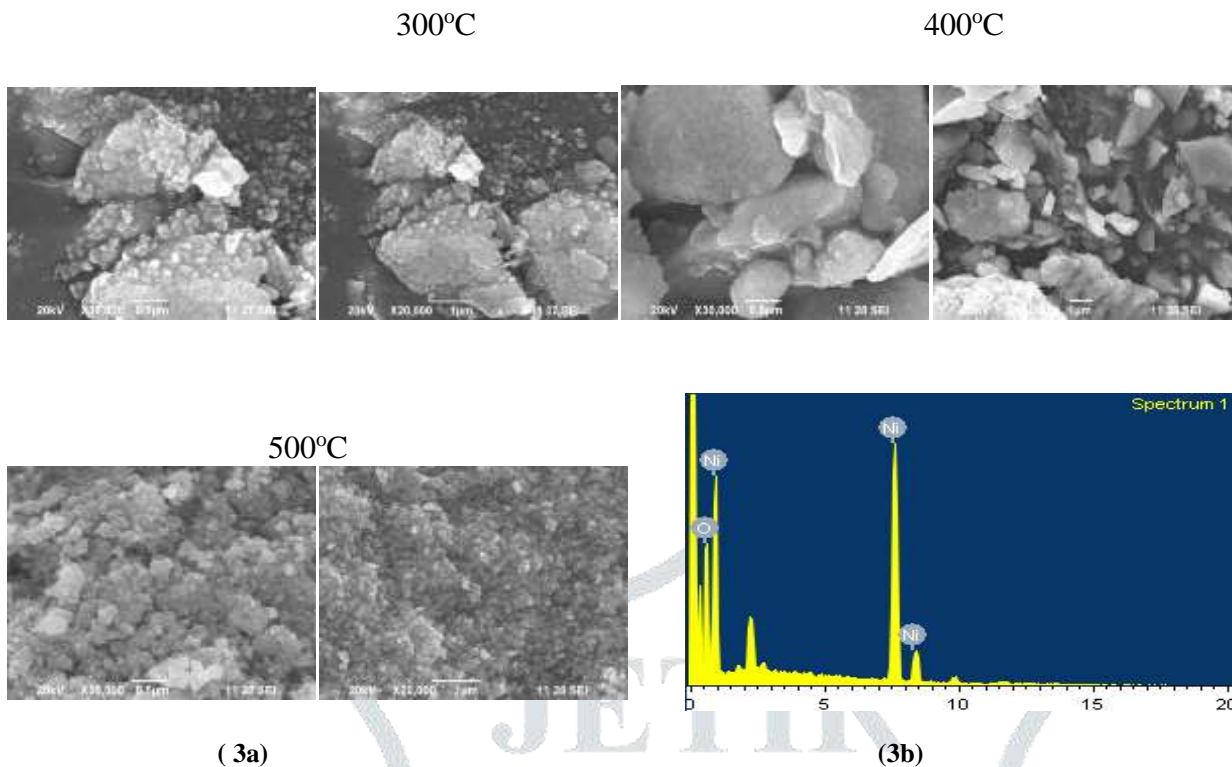


Fig. 3.(a) SEM and (b) EDX spectrum image of NiO nanoparticles.

Table 1-Data of Energy Dispersive X-ray spectroscopy (EDAX) pattern From NiO nanoparticle

Element	Weight %	Atomic %
O K	43.98	74.24
Ni K	56.02	25.76
Total	100	

3.3 FUNCTIONAL ANALYSIS:

FTIR spectroscopy is a useful tool to investigate functional groups, modes of vibration and behaviour of nickel precursor. Fig. 5 shows the FT-IR spectrum of NiO- sample.

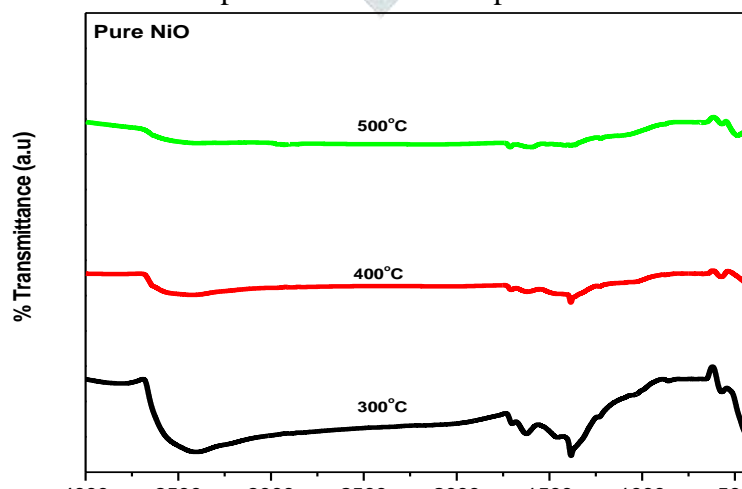


Fig. 4: FTIR spectrum of NiO nanoparticles annealed at 300°C , 400°C and 500°C for 1 hr

The peak around 3404 cm^{-1} , 3424 cm^{-1} and 3410 cm^{-1} on the FT-IR spectrum is related to O-H stretching bond. The absorption at 1623 cm^{-1} , 1620 cm^{-1} and 1592 cm^{-1} is attributed to hydroxyl groups. The absorption bands at 1454 cm^{-1} , 1384 cm^{-1} , 1363 cm^{-1} , and 1229 cm^{-1} indicate the existence of carbonates and the band at 2883 cm^{-1} and 1712 cm^{-1} correspond to C-H stretching mode [13], as shown in Fig 4. The spectrum also contains strong absorption bands at around 433 cm^{-1} , 435 cm^{-1} , and 485 cm^{-1} associated to Ni-O vibration band [14]. The above information confirmed formation of pure NiO nanoparticles.

3.4 VSM Analysis of Sample

The magnetic measurements of pure NiO nanoparticles were performed by a vibrating sample magnetometer (VSM model 7402) technique at Maximum Magnetic field 2.5 T at room temperature. This is shown in Fig 5. Pure NiO nanoparticles depict almost linear response to the applied magnetic field. In addition, both loops pass through the origin [15] and show antiferromagnetic behavior of NiO nanoparticles.

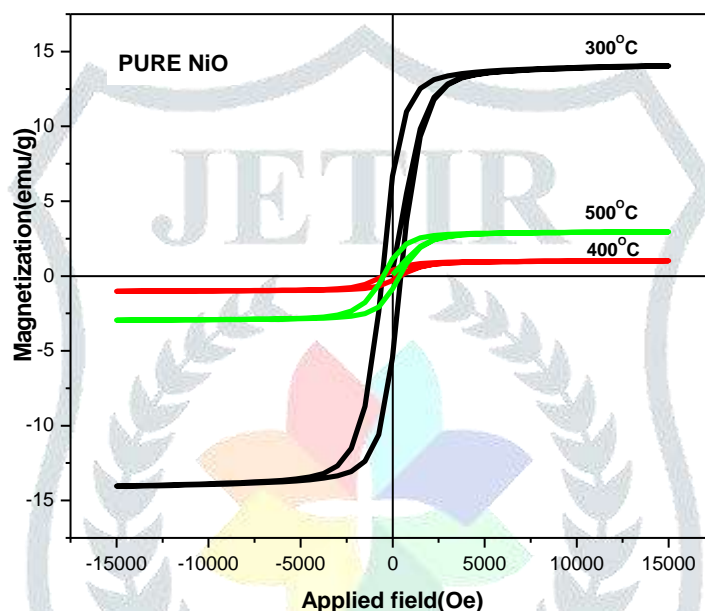


Fig. 5: M–H loops for pure NiO nanoparticles annealed at 300°C, 400°C and 500°C for 1 hr.

The saturation magnetization (M_s), the remnant magnetization (M_r), the coercivity (H_c), and squareness ratio (S) ($S = M_r/M_s$) are listed in Table 2. The tabulated data show that coercivity H_c decreases with the increase of temperature, indicating that H_c is a function of the particle size. This variation of H_c with particle size can be explained on the basis of domain structure, critical size, and surface as well as interface anisotropy of the crystal. A crystallite will spontaneously break up into a number of domains in order to reduce the large magnetization energy if it is a single domain [16]. From Table 2, it is clear that $SQR < 0.5$ for all samples indicating that the particles interact by magneto static interaction and the anisotropy decreases in crystal lattice [17,18]. The SQR for different temperatures of NiO nanoparticles are found to be below 0.5 indicating that the pure NiO do not have the properties of recording medium [19]. The prepared materials can be used as soft electro-magnetic materials in constructing transformers and cathode materials in batteries.

Table 2-Magnetization (M), magnetic field (H) of the samples (NiO Nanoparticle).

Parameters	300°C	400°C	500°C
Size (nm)	7	12	20
M_r (emu/g)	6.0654	0.3406	2.1136
M_s (emu/g)	14.0397	1.0177	6.2244
H_c (G)	500.35	469.52	440.53
SQR	0.4320	0.3347	0.3395

4. CONCLUSION

The present study reported on Nickel oxide nano particles has been successfully synthesized by solvo thermal method (nickel acetate and ethylene glycol). The synthesized nanoparticles were characterized by XRD, SEM, EDAX, FTIR and SEM Analysis. The results obtained from crystallite size from XRD and SEM images confirm the nanocrystalline nature of the synthesized product. The VSM measurement of nickel oxide nanoparticles indicates the room temperature ferromagnetic behaviour of NiO nanoparticles. The prepared nano particles can be used as soft electro-magnetic materials in constructing transformers and cathode materials in batteries.

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