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Advances in Magnetic Properties of NiO Nano-Particulates with Calcinations Temperature

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The materials have peculiar advance properties with increase of surface area and have optimum advancement at miniaturization at nano scale (1-100 nm) .The Nickel Oxide (NiO) nano-particulates have significant properties at nanoscale in the field of MRI, fabrication of chips, electrodes formation and super-capacitors. In present work, the researchers synthesized the NiO nano-particulates with high efficiency via advance chemical co-precipitation techniques and thereafter, the samples were calcined at 200°C, 400°C, 600°C & 800°C for constant time 2 h. The calcined powdered samples were examined through various characterization tools such as X Ray Diffraction (XRD), Fourier Transform Infra Red spectrum (FTIR), Field Emission Scanning Electron Microscopy (FESEM) & Vibrating Sample Magnetometer (VSM) etc. The XRD spectrum examination have concluded that the calcined NiO samples were face centered cubic in formation with crystallite size 34.88 nm, 19.48nm, 36.11nm and 36.35nm at 200°C, 400°C, 600°C & 800°C, respectively. The IR spectrum of samples reflects that O-Ni-O peaks were found at position 505cm^{-1} and shows slightly shifting with blue shift with increase of calcinations temperature. The FESEM magnified images of NiO samples calcined at 600°C for 2 h were truncated spherical in shape and agglomerated in nature. The Vibrating Sample Magnetometer (VSM) were deployed to examine the variation in magnetic properties of samples with temperature and finding of study proved that samples were ferromagnetic in nature with small hysteresis loss and magnetic susceptibility (χ) increases from (χ=1.31X10^{-}3) 200°C to (2.53x10^{-6}) 600ºC and thereafter, decreases with further increase of temperature at (2.22x10^{-6}) 800°C.

Keywords: Nickel Oxide nano-particulates, XRD, FTIR, FESEM, VSM

1 Introduction

Since many years ago, nanotechnology has been the subject of extensive and cutting-edge field of research. Firstly, the Richard Feynman introduced this technology in 1959 and summarized that particles with sizes in the nano meter range were related with nanotechnology[1-2]. Nanoparticles (NPs) produced by metals such as Cu, Zn, Mg & Fe have distinctive properties based on the shape and size of NPs[3-4]. Owing to the tiny quantum tunnel effect and enhanced surface to volume (S/V) ratio, it is expected that nano-crystalline NiO would have many more applications, including the fabrication of different magnetic materials, gas sensors, catalysts, electro chromic films, and battery cathodes also NiO semiconductors are inexpensive to utilize as energy storage materials, by possessing all these type of importance it become a compelling research topic for today researchers[5-7]. By keeping in mind, the researchers created their attraction towards synthesis of NiO by advanced chemical root method and thereafter, calcined the samples at various calcination temperatures. The powdered calcined samples were characterized by various tools such as X Ray Diffraction(XRD), Fourier transform Infrared (FTIR) spectrum and Vibrating Sample Magnetometer(VSM).The newer materials NiO calcined at 600°C exhibits its superiority in respect of saturation field whereas, the NiO calcined at 800°C were enhanced corrective field over a wide range of magnetic field[8]. The novel NiO powdered samples have ferromagnetic in nature over a wide range of temperature and saturation applied field. The results were discussed in this paper.

2 Experimental

2.1 Synthesis of NiO Nano Particulates

There are many ways of formation of NiO nano particsles, out of which microwave irradiated chemical co-precipitation method is very simple and easy for the synthesis of NiO nanoparticles. Here the

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*Corresponding author: E-mail: sheelarohilla151@gmail.com author declares that all the chemicals used were in A.R. grade and not purified at laboratory scale. Taking appropriate concentration of $NiCl_2.6H_2O$ dissolve in 100 mL distilled water then added 2.0 M NaOH solution as precipitating agent until or unless the pH value of the solution reached to 9.0 with dis association of ions takes place and greenish color ppt were formed. The precipitates were kept for 24 h to stabilize the process. Thereafter, filtered and washed the precipitate with ethanol solution. Then resulted filtered cake put into microwave oven for 15 min at 250°C for dehydration and further calcined it at the different temperatures such as 200°C, 400°C, 600°C and 800°C for 2 h. The calcined samples were crushed in aged mortar until it becomes powder and then stored in vaccum bottle for further characterizations techniques.

2.2 Characterization

The various calcined samples were analyzed using an X-ray diffractometer (Perkin Elmer) with CuKα (1.5406A°) radiation with diffraction angle 2θ in range of (10°-80°) and to determine the structural properties of the resultant materials respectively. A Vibrating Sample Magnetometer was used to measure the magnetic properties of the samples, and a Diamond ATR & Pellet accessory (Perkin Elmer) was used to measure the Fourier transform inferred (FTIR) spectrum in the range of 400 cm^{-1} to 4000 cm^{-1}.

3 Results and Discussion

3.1 XRD Analysis

The X Ray diffraction technique was used to study the crystallographic behavior and effect of calcinations temperature on the pure NiO nano particulates. A graph between XRD peak positions (2θ) and intensity of diffracted X-ray beam was plotted which displays the X-Ray diffraction spectra of pure NiO nanomaterials calcined at 200°C, 400°C, 600°C and 800°C each for 2 h (Fig.1). The scanned data of calcined samples of Nickel Oxide Nano particulates at various temperature for a fixed duration of 2 h were analyzed in graphical representation as shown below:-

At 200°C the XRD spectrum shows the most intense peak at 2θ≈31.7° which might be due to the resultant of $Ni(OH)_2$ and NiO crystalline as both the phase occurred at this temperature. The occurrence of NiO peaks were also supported by IR spectroscopic peaks at wave number 505 cm^{-1}. According to the XRD spectrum the hexagonal closed packed (hcp) structure were in creation and corresponds to $Ni(OH)_2$ formation corresponding to $2\theta \leq 20^{\circ}$. However, no such $Ni(OH)_2$ peak was found at temperature above 200°C for 2 h and above 200°C the most intense peak was attributed at more or less constant 2θ≈ 43.3° which corresponds to the FCC structured NiO (JCPDS File.47-1049) with diffraction peaks corresponding to planes (111), (200), (222), (311) and (220) supporting the FCC structured NiO NPs [9-10]. However, the Debye Scherer formulation shown in Eq 1 was taken in consideration for calculation of crystallite size.

$$D_{hkl} = \frac{0.9\lambda}{\beta_{hkl}\, Cos\theta} \quad \text{... (1)}$$

where β_{hkl} is the line's full width at half maximum (FWHM), λ is the wavelength of CuKα radiation (λ=1.5406A°) & θ is angle of diffraction[11].

The recorded data of XRD parameters such as peak position corresponding to maximum intensity, Full width at half maxima (FWHM) and crystallite size calculated by taking most intense peak in consideration were tabulated in Table 1.

The tabular data reflects that the major peak at 2θ≈31.7° signifies the formation of Ni Hydroxide with NiO nano-particulates at 200°C. However, the most

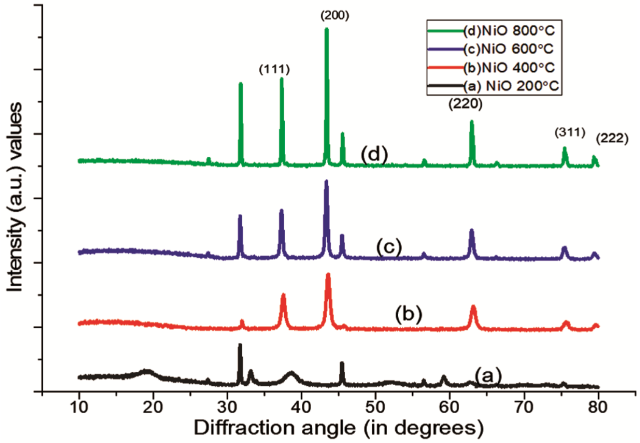

Fig.1 — XRD spectrum of NiO nano-particulates calcined at (a) 200°C (b) 400°C (c) 600°C, and (d) 800°C for 2 h each

Table 1 — XRD data of NiO Nano crystalline materials calcined for 2 h and at temperature 200°C, 400°C, 600°C & 800°C

Sr. No	Calcination temperature in °C	Peak position(2θ) (in degree)	FWHM (in radian)	Size (in nm)
1	200	31.7	0.234	34.88
2	400	43.5	0.434	19.48
3	600	43.2	0.234	36.11
4	800	43.2	0.267	36.35

intense peak position shift for temperature 400°C, 600°C & 800°C at less or more constant $2\theta \approx 43.3^{\circ}$ which were attributed by the formation of NiO FCC in major proportion with increasing order of size of nanoparticles however, $Ni(OH)_2$ peak were disappeared at temperature higher than 200°C[12].

3.2 IR Spectrum Analysis

The FTIR spectrum was used to scan the samples for detection of groups or contaminants present in the samples of the NiO NPs after calcined at different temperature. The spectrum shows the variation of transmittance percentage versus wavenumber of radiations as shown in Fig. 2.

The broad band noticed nearly at wavenumber 3600 cm^{-1} and were attributed to OH vibrations whereas, the additional sharp peak at 3633 cm^{-1} in Fig. 2 (a) clarify the formation of $Ni(OH)_2$ in samples. The peak detected at 1674 cm^{-1} is attributed to water molecules bending vibration[5,13]. The O-C=O symmetric and asymmetric stretching vibrations and the C-O stretching vibration resulting from the adsorption of ambient CO_2 or ethanol are responsible for the peaks detected at 1350 cm^{-1}, 1300 cm^{-1}, respectively. A strong peak at 505cm^{-1} corresponds to Ni-O vibration[14-15]. The IR spectrum findings supported XRD results.

3.3 FESEM Analysis

The FESEM tool JEOL JSM 7610F Plus is used to see the magnified view of nanoparticles and applied under operating conditions of voltage 20.0 KV with magnification rate x5000.The FESEM magnified images of NiO samples calcined at 600°C for 2 h were truncated spherical in shape and agglomerated in nature. The scanned data of FESEM is shown in Fig. 3.

3.4 VSM Analysis

The vibrating sample magnetomete were used to examine the magnetic behavior of various calcined samples. The external magnetic fields were applied within range of -3000(Oe) to +3000(Oe) and corresponding intensity of magnetization were recorded to form a B-H curve. The VSM results are hereby plotted in form of graph which shows the variation of applied magnetic field intensity with intensity of magnetization as shown in Fig. 4.

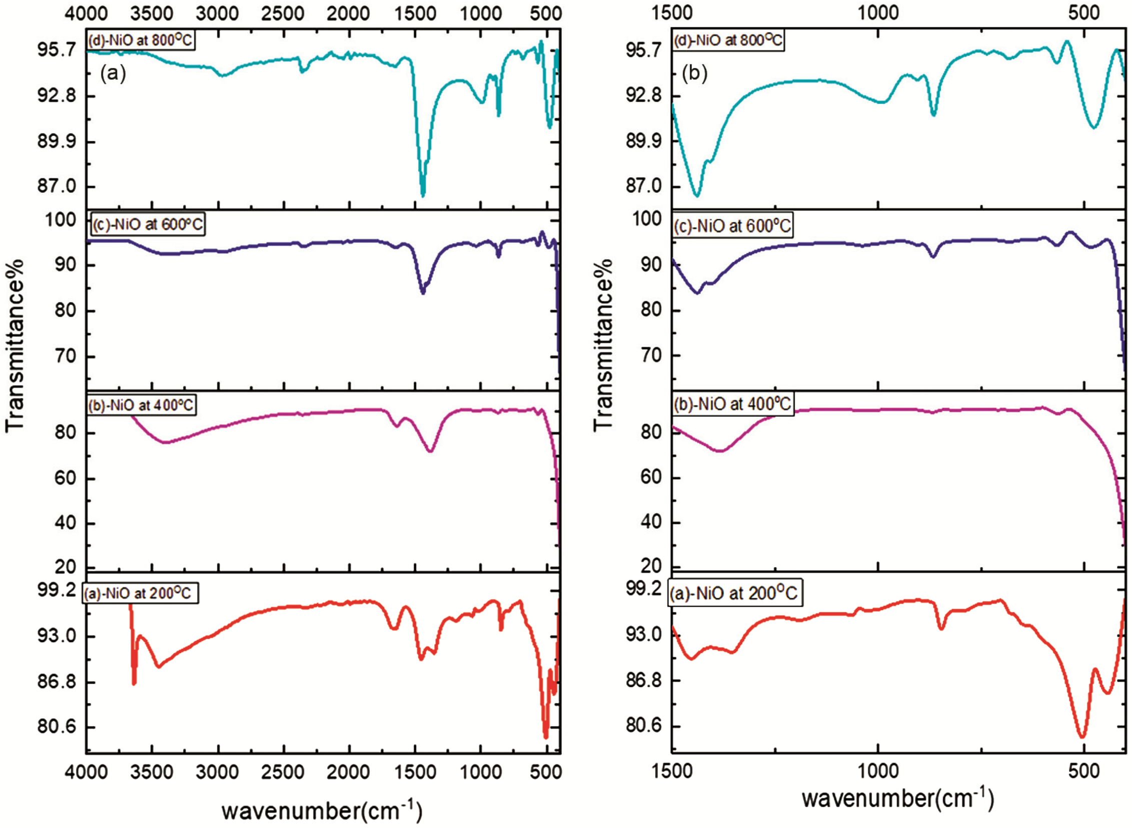

Fig. 2 — (a) IR Spectrum of NiO NPs calcined at (a) 200°C,(b) 400°C,(c) 600°C,(d) 800°C for 2 hours respectively (b) Magnified view of Fig. 2 (a).

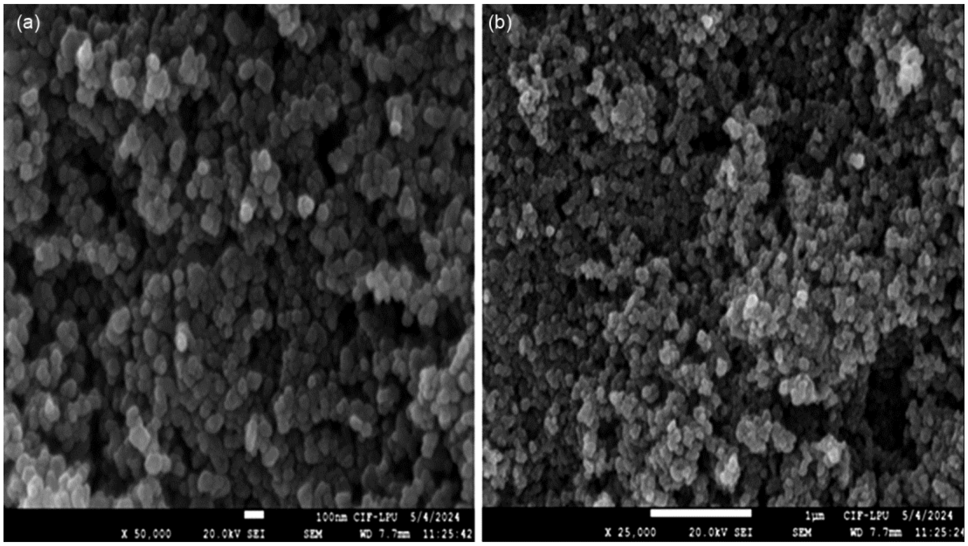

Fig. 3 — FESEM images of Pure NiO at 600 ºC at different magnification conditions of scanning microscope

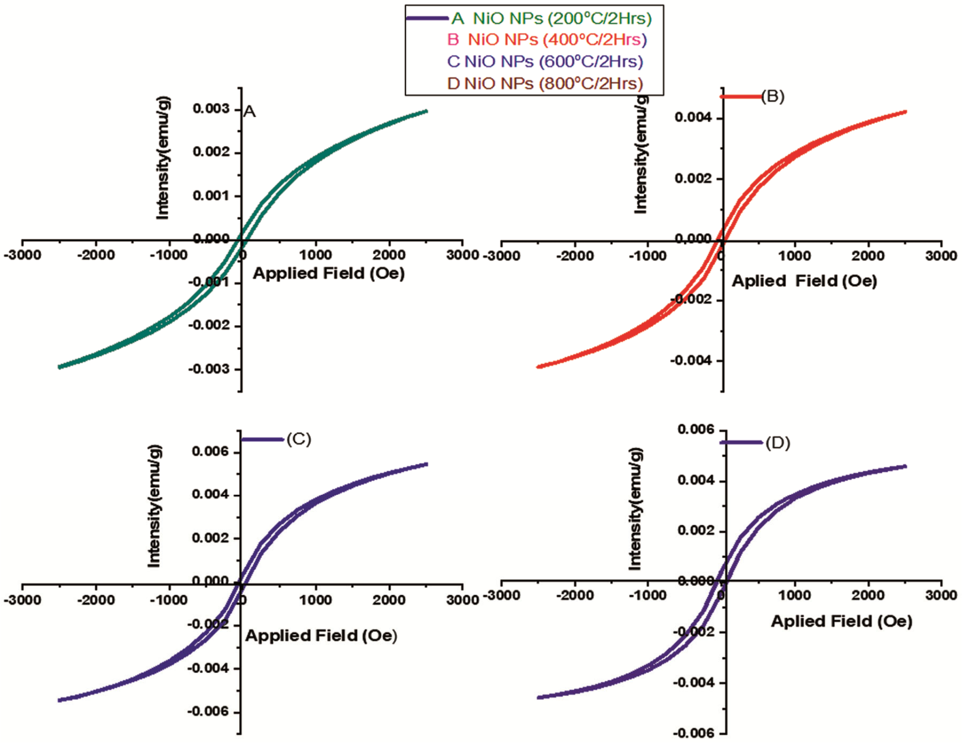

Fig. 4 — VSM graph of NiO nano-particulates calcined at (a) 200^{o}C (b) 400^{o}C(c) 600^{o}C and (d) 800^{o}C/2h

The graphical data shows that the intensity of saturated magnetization increases with increase in temperature from 200^{o}C to 600^{o}C however, intensity decreases when temperature further increases from 600^{o}C to 800^{o}C and also magnetic susceptibility (χ) increases from (χ=1.31×10^{-3}) 200^{o}C /2 h to (2.53×10^{-6}) 600^{o}C/2 hrs and thereafter, decreases with further increase of temperature at (2.22×10^{-6}) 800^{o}C/2 h which might be due to achieving Neel temperature of crystal and resulted material of NiO at temperature 600^{o}C and above shows paramagnetic behavior[16-18].

The conclusive data of various magnetic parameters at different calcination temperature such as Maximum saturation intensity, coercive field,

Table 2 — The hysteresis parametric study of pure NiO nanoparticles calcined at different temperatures

S. No.	Sample NiO pure calcined at	Ms. Saturation (emu/g)	Coercive (Hc Oe)	Remnant (emu/g)	Squareness factor	M at Max field (emu/g)	Energy Loss(Oe)	Saturation Applied H_s (Oe)
1	200°C	2.95E-3	58.68	164.75E-6	0.056	2.94E-3	8.11E+0	2256.84
2	400°C	3.51E-3	38.55	214.5E-6	0.008	3.50E-3	8.51E+0	2135.23
3	600°C	5.451E-3	46.758	305.97E-6	0.056	5.45E-3	9.66E+0	2151.47
4	800°C	4.580E-3	67.554	426.228E-6	0.093	4.57E-3	9.12E+0	2057.26

remnant, squareness factor, Intensity of magnetization at maximum field, energy loss & saturation applied field were tabulated in Table 2.

The tabular data reflects that the Maximum saturation intensity is $2.95E^{-3}$ at 200°C, $3.51E^{-3}$ at 400°C and $5.45\ 1E^{-3}$ at 600°C which shows increasing trend with temperature however, it decreases beyond 600°C i.e. $4.58E^{-3}$ at 800°C.

4 Conclusion

The pure NiO nano particulates were successfully synthesized via microwave irradiated chemical co precipitation method and further the effect of calcination temperature were analyzed for fixed duration 2 hours. The XRD results concluded that the $Ni(OH)_2$ Nano particulates were in formation FCC crystallite size of 34 nm at 200°C and NiO nano particles were formed with sizes 19.48 nm, 36.11 nm & 36.35 nm at temperature 400°C, 600°C & 800°C respectively. The VSM study reflects that the intensity of saturation increases with temperature from 200°C to 600°C however, beyond 600°C the saturation intensity decreases. So study concluded that the spin movements of the particles were highly dependent on calcination temperature. The newer calcined material may have futuristic material for applications of electromagnet and memory chips formation. The electron microscopic image of samples recorded at scale of 100nm shows that truncated spherical like crystalline and agglomerated in nature.

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