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Modifications in Magnetic properties with Copper Oxide dopant concentration in $NiCuO_{2\pm\delta}$ Nanocrystalline

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In present work, the Copper doped Nickel Oxide nanoparticles with molar concentrations (0%, 5%, 10% & 20%) were synthesized by microwave irradiated chemical co precipitation protocol and thereafter, calcination, the required nano-crystalline materials samples were examined through various techniques in order to make a comparative analysis of the magnetic behavior of pure Nickel Oxide nano-crystalline with various Copper doped Nickel Oxide nano-crystalline materials. The structural outcomes from X-Ray Diffractometer (XRD) tool and Fourier Transform Infar Red (FTIR) technique shows that FCC crystalline structure of NiO occurs. The crystallite size was determined using Debye Scherer formula and average crystallite size was 36.35nm for un-doped NiO nano particles whereas on addition the content of doping the crystallite size varies from 37.38nm for Cu 5%, 36.10nm for Cu 10% and 28.07nm for Cu 20% at when calcined at 600 °C temperature for 2 hours respectively. The IR spectrum peaks at positions 478 cm-1were attributed by O-Ni-O vibration whereas peak at 567 cm-1and 687cm-1 were assigned to O-Cu-O vibrations of CuO & Cu2O crystalline respectively.VSM results of various calcined samples with different dopant concentrations reveals that the behaviors of nanoparticles were ferromagnetic in nature with small hysteresis loss. However, the remarkable changes were noticed in saturated magnetization intensity with Cu2+ ion concentration in NiO lattice structure. The Cu 10% concentration doped NiO nanostructure materials were recommended as key materials for electromagnetic formation for MRI equipment applications with high range of applied magnetic field.

Keywords: NiO nano structured materials; XRD; FTIR; FESEM; VSM

1 Introduction

Over the past two decade, nanotechnology has provoked a lot of interest in nano sized particles with size ranging from one to one hundred nanometers and assembly may be consist of metals, organic materials, metal oxides or carbon Nano tubes *etc*.[1]. The nanoparticles exhibit unique Physical, Chemical and Biological characteristics at the nanoscale as compared to their bulky size materials. Numerous features, including greater mechanical strength, enhanced reactivity or stability in a chemical process can be ascribed to this occurrence[2]. The past work shows that the enhanced magnetic and electrical properties with long life cycle utilities of Nickel oxide nano-crystalline were proving their key role in making of technological application in various fields such as super capacitor, MRI, transformer core and electrodes of cell[3-6]. Whereas CuO nanocrystalline have significant application in the field of bio-sensor, antibacterial, electrical & magnetic field[7-9]. In present work, the microwave irradiated chemical co-precipitation technique were employed to synthesis Copper incorporated NiO nanoparticles with various concentrations such as 0%, 5%, 10% & 20% respectively. The as synthesized samples were calcined at temperature 600 ^{o}C for 2hrs for improving crystallinity. The calcined samples were scanned through X-Ray diffraction (XRD) tools and the results confirmed the existence of Cu^{2+} at NiO lattice site in FCC crystalline. The Vibrating Sample Magnetometer (VSM) data were used to produce Hysteresis loop between Magnetization Intensity M_H and applied magnetic field intensity H also the observations taken from hysteresis loop were helpful to estimate the Residual intensity, Coercive field, Saturation induced field, squareness factor, Saturated applied magnetic field, Energy loss/cycle and magnetic susceptibility of specimen over a wide range of applied magnetic field intensity. The morphology and texture of nano-particulates were examined by Field Emission

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Scanning electron microscopy (FESEM) and FESEM images show that specimen particles were nano sized in scale and flake like morphology with agglomerated in nature[10-12]. The newer CuO 5% incorporated NiO nanoassemblies can have significant candidature in formation of permanent magnet[13].

2 Experimental

2.1 Synthesis Mechanism

The Researcher declares that the entire chemicals taken in present work were analytical grade (i.e. 99.9% pure) and no further purifications were made at laboratory scale. NiO nano-particulates were synthesized by microwave irradiated chemical co-precipitation protocol by taking 1.0 Molar concentration of ($NiCl_2.6H_2O$) and appropriate amount of $Cu(NO_3)_2.3H_2O$ in 100 ml doubly de-ionized (DDI) water and copper salt concentration increases with increase of dopant concentration 0%, 5%, 10% & 20% respectively. The resulted bluish-green transparent solution were transformed in precipitation with addition of 2.0M NaOH solution at about ph ≈9.0 and precipitate were stabilized at bottom after 24 hours aging process then filter it by WattMann filter paper. The filtered cakes were multiply washed by diluted ethanol & DDI for removal of the extra contaminants occurred Cl^-ions and NO_3^-ions impurity and resulted cake was dried in the microwave oven at 150 ºC for quarter an hour in two shifts. Furthermore, the subsequent samples were placed in muffle furnace for calcination at 600 ºC for two hours and grinded it in agate mortar until it becomes fine powder. The resulted NPs powder samples were used for characterization techniques and stored samples in evacuated sample holders.

2.2 Characterization

The basic structural parameters of various CuO incorporated calcined samples were studied with the help of X-ray diffractometer with CuKα (λ=1.5406A°) radiation with diffraction angle 2θ in the range of (10°-80°). The Magnetic characteristics of calcined samples were assessed by using a Vibrating Sample Magnetometer M_HVs. H (±1.5 T) whereas elemental group were analyzed by Fourier Transform Infra Red (FTIR) spectroscopic tools Diamond ATR & Pellet attachment (Perkin Elmer). The morphology of sample were determined by instrument JEOL Field Emission Scanning Electron Microscope. The results of study were discussed in next section of paper.

3 Results and Discussion

3.1 XRD Results Analysis

The X-Ray Diffraction technique was sited to examine the crystallographic study of various calcined samples (600 ºC for 2 hrs.) of NiO pure and CuO doped NiO with CuO concentration were taken 5%, 10% & 20% to see the influence of dopant concentration on crystallographic modification within range of 2θ≈ 10° to 80° & the results of study were shown in Fig. 1 and calculated crystallite size were represented in Table 1.

The exhibitance in Fig. 1 showed that peak position at 2θ ≈ 29° , 31.3°, 37.4°, 43.3°, 45.6°, 51.5°, 56.5°, 62.87°, 66.3°, 75.4°, 79.4° were noticed and among these the peaks at 2 θ ≈ 31.3°, 37.4°, 43.3°, 45.6°, 56.5°, 62.87° and 75.4° corresponds to JCPDS card-47-1049 of NiO nano crystalline whereas peak occurred at 29°, 51.5° was corresponds to Cu_2O and peak at 66.3° be corresponds to CuO analogous to JCPDS card 05-0667 and JCPDS card 45-0937 of Cu_2O and CuO respectively. The most intense peak appears at 2θ ≈ 43.3° which is more or less constatnt for NiO for various doping concentration, which give a clear indication of existence of NiO phase with FCC structure. The average crystallite size (D) determined by Debye Scherer formula

$$D=\frac{0.89\lambda}{\beta Cos\theta} \quad \text{... (1)}$$

Where λ is wavelength of X-Rays, β is Full width at half maxima (FWHM) and θ is Bragg's diffraction angle and calculated crystallite sizes were précised in Table 1.

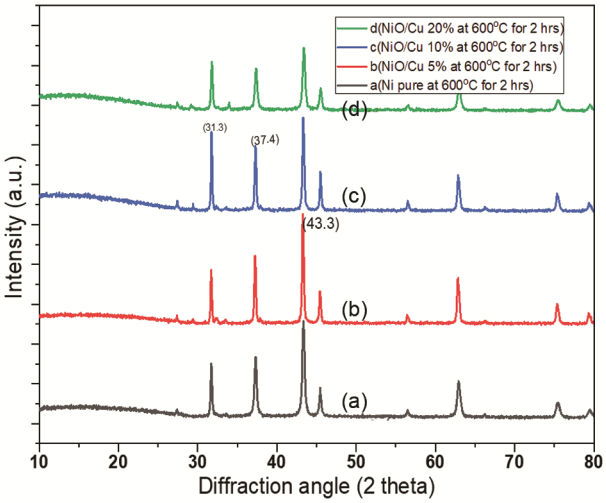

Fig. 1 — XRD Spectrum of calcined samples of CuO- NiO nano-crystalline with Copper concentration (a) 0% (pure NiO), (b) 5%, (c) 10% & (d) 20% at calcination temperature 600 °C for 2 hrs respectively

Table 1 — The Crystallite size of NiO pure and CuO doped NiO nano crystalline calcined at 600 ºC for 2 hrs.

Sr. No	Sample Name	FWHM (in radian)	Most intense peak position 2θ (in degree)	Crystallite size (in nm)
1	NiO pure	0.334	43.30	36.35
2	NiO+CuO5%	0.226	43.27	37.38
3	NiO+CuO10%	0.267	43.31	36.10
4	NiO+CuO20%	0.287	43.31	28.07

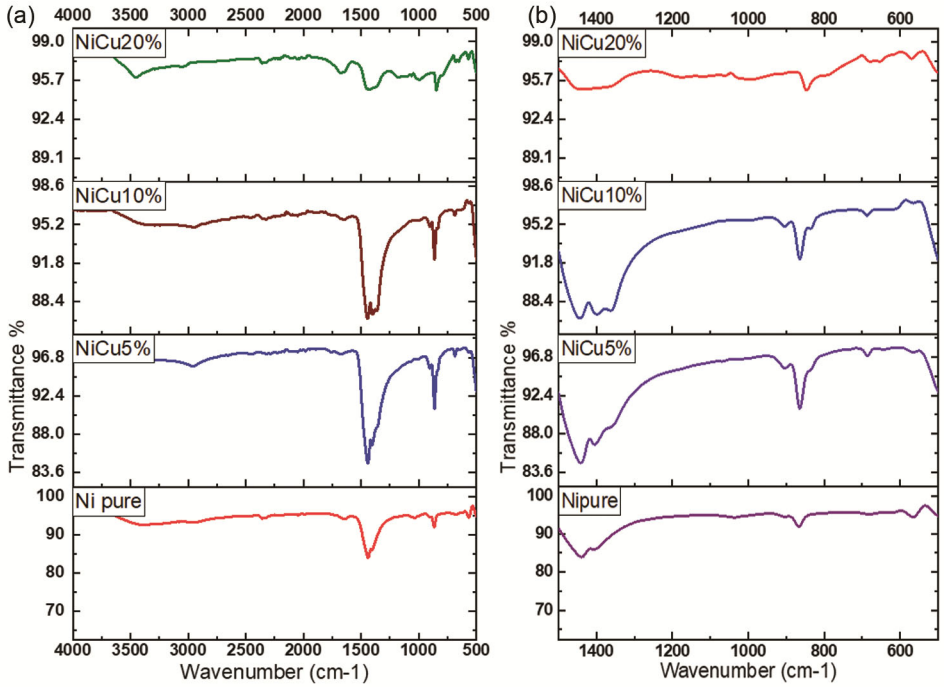

Fig. 2 — IR Spectrum of calcined samples of CuO- NiO nano-crystalline with Copper concentration (a) 0% (pure NiO), 5%, 10% & 20% at calcination temperature 600 °C for 2 hrs. (b) Magnified view of (a)

The perusal XRD parameters & spectral analysis shows that the average crystallite size decreases with increases of dopant concentration CuO from 5% to 20% i.e. 37.38 nm to 28.07nm respectively. However, small increment in crystallite size were noticed in 5% Cu concentration in NiO with NiO pure nano crystalline and it might be due to crystalline defects or distortion occurred with small concentration of CuO at NiO lattice sites.

3.2 FTIR Spectroscopic Results Analysis

The IR spectroscopic results were used to study the metal oxide groups and other entities present in calcined samples. The transmittance rates were recorded with variation of wavenumber which lies in the range from 400cm^{-1} to 4000cm^{-1}. The IR spectrum of NiO nanoparticulates with various dopant concentrations of CuO has been shown in Fig. 2.

The perusal of Fig. 2 shows that two broad bands occurred at 3482 cm^{-1} and 2373cm^{-1} attribute the OH vibration of H_2O molecules present in atmosphere of samples[14-15]. The sharp IR peak at position 478cm^{-1}were attributed by O-Ni-O vibration whereas peak at 567cm^{-1} and 687cm^{-1} were assigned to O-Cu-O vibration of CuO & Cu_2O crystalline respectively. Moreover, the peaks occurred at 1439 cm^{-1} and 1385cm^{-1} attribute by O=C=O vibration of CO_2 molecules.

3.3 FESEM Image Analysis

The pure Fig. 3 NiO samples calcined at 600 ºC for 2 hours were scanned by microscopic tools FESEM under the operating condition of voltage 20.0 KV and with magnification rate 50,000 with scaling of micrograph 100nm. The sample images reveals that the morphology of pure NiO were nanoflakes with agglomerated form but CuO 10% doped NiO specimens were 2D disc ortablet in formation[16].

3.4 M-H Curve Analysis

The Vibrating Sample Magnetometer (VSM) tool was used to analyze the magnetic assets of various calcined samples. The magnetic properties of

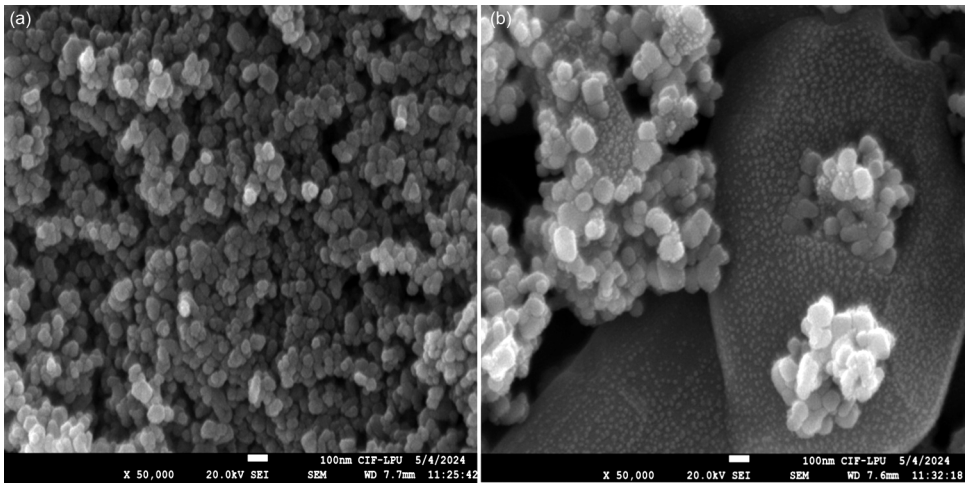

Fig. 3 — FESEM image of sample calcined at 600 °C /2hours (a) Pure NiO (b) CuO 10% incorporated NiO specimen

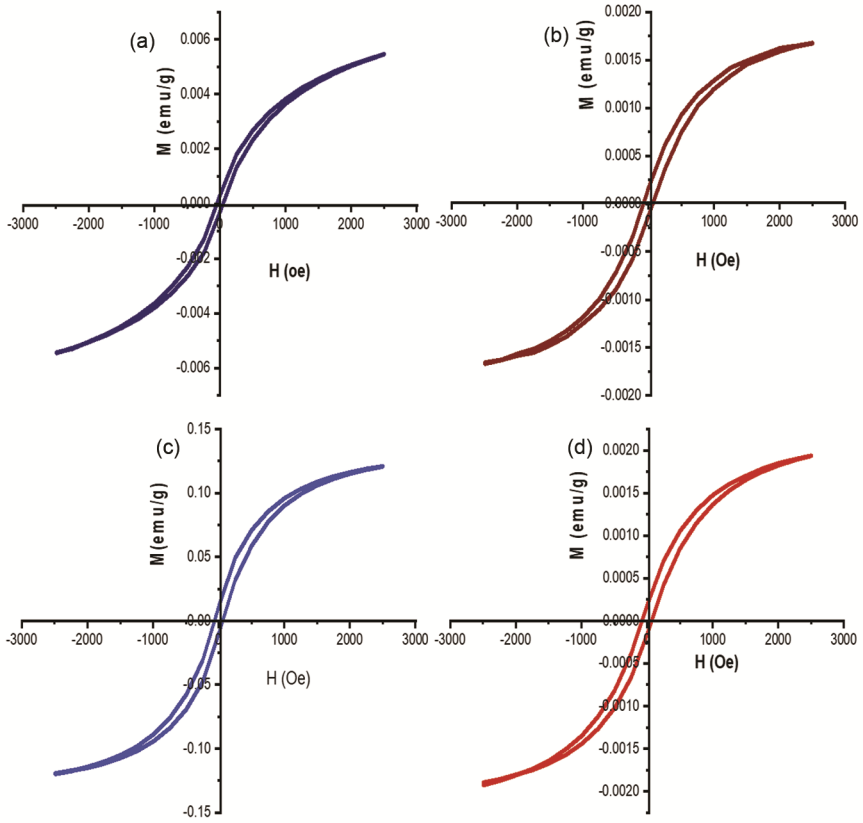

Fig. 4 — The VSM spectrum of NiO nanoparticles with various CuO concentrations (a) 0% CuO, (b) 5% CuO (c] 10% CuO & (d) 20% CuO

powdered samples of pure NiO and CuO (5%, 10% & 20%) incorporated NiO samples calcined at 600 ºC /2 hours were examined through VSM tools at room temperature and over a wide range of applied magnetic field intensity H ±3.0 T .The first instance of M-H curve in Fig. 4 showed that the newer calculated samples were ferromagnetic in apprearance with significant variation in saturation intensity of magnetization. The various calculated parameters were tabulated in Table 2.

The perusal of above tabular data reflects that the newer 5% CuO doped NiO materials have remarkable variation in residual intensity in respect of pure NiO and 10% & 20% CuO doped NiO nano crystalline.

Table 2 — The Hysteresis parametric study of various concentrations (5%, 10% & 20%) of Cu dopant in NiO nanoparticles calcined at 600 °C for 2 hours.

Sr. No	Sample	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	NiO pure	$5.451x10^{-3}$	46.758	30.597x10-6	0.056	$5.45x10^{-3}$	$9.665E^{-0}$	2151.47
2	NiO +CuO(5%)	$1.672x10^{-3}$	70.957	142.974	0.086	$1.66x10^{-3}$	$7.229E^{-0}$	2043.45
3	NiO +CuO(10%)	$120.22x10^{-3}$	61.718	$10.456x10^{-3}$	0.087	$120.22x10^{-3}$	$7.764E^{+0}$	1964.12
4	NiO +CuO (20%)	$1.932Ex10^{-3}$	72.498	$166.78x10^{-3}$	0.086	$1.91x10^{-3}$	$7.462E^{+0}$	2068.85

So, therefore 5% CuO doped NiO nanocrystalline might have reportable properties in formation of permanent magnet over a wide range of applied magnetic field (H). Moreover, 10% CuO doped NiO responds significantly over the saturation field *i.e*.$120.22X10^{-3}$emu/g with lowest remittance rate in comparison to 5% & 10% CuO doped NiO nano crystalline so the newer material may be recommended for formation of electromagnet based applications[17].

4 Conclusion

The pure NiO & various CuO doped NiO nanocrystalline were successfully prepared via microwave irradiated chemical co-precipitation protocol and therefore calcined at 600 ºC for 2 hours for crystalline formation .The XRD results concluded that crystallite size decreases with increases of CuO concentration (5%, 10% & 20%) in NiO lattice site of Face Centered Cubic structure .The IR spectrum absorption peak at about 478 cm^{-1},567cm^{-1} & 687cm^{-1} were confirmed the CuO incorporation in NiO crystalline .The electron microscopic images of pure NiO & CuO(10%) doped NiO were taken for comparison and reflects 2D tablet formation with contrast. The magnetic properties of samples proved the suitability of 5% CuO doped NiO sample proposed material as permanent magnet ,whereas 10% CuO doped NiO, material have key featured properties as a electromagnet over a wide range of applied field intensity.

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