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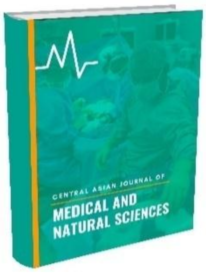

Structural, Optical, and Magnetic Properties of Nickel Oxide Nanoparticles Through Precipitation Method

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ABSTRACT: Nickel oxide nanoparticles (NiO NPS) have been prepared via chemical precipitation method. In this work, we are studying about the structural, morphological, and magnetic properties. The NiO NPS are characterized by different ways like XRD, SEM/EDX, FTIR, Antibacterial and VSM analysis. XRD indicates that the FCC structure of NiO NPS, with the crystallite size from 17.89 nm to 52.37 nm. The spherical shape was observed in SEM image. The basic composition of NiO NPS is determined by using the EDX. FTIR gives two wide peaks appeared at 421and 481 cm^{-1} due to Ni–O stretching vibration clearly indicates the formation of NiO at 600°C and 800°C. The VSM results demonstrate that the NiO NPS showing weak ferromagnetic behavior at 600 °C and 800 °C temperature.

Key words: NiO NPS; precipitation method; XRD; FT-IR; VSM.

Introduction

Recently, investigated the most important inorganic material is nickel oxide nanoparticles (NiO NPs). The NiO is an essential material that can be widely used in many applications, such as capacitor, solar cell, and rechargeable lithium ion batteries. The NiO is one of the p-type semiconductor materials with wide band gap of 3.6-4 eV [1]. The magnetic nanoparticles are most widely utilized in conducting paints [2], rechargeable batteries [3], chemical catalysts [4], optoelectronics [5], magnetic recording media [6], Ferro-fluids [7] and drug delivery so on. In addition, NiO NPS have attracted and great attention because of their thermal applications and their specific chemical and physical properties [8]. Among the various nanomaterials, NiO nanomaterials exhibit significantly chemical, electronic, thermal, mechanical, magnetic, catalytic properties and optical properties in comparison with their bulk materials and have been extensively attracted application. Nowadays the pathogenic bacteria cause many diseases in human beings. NiO nanoparticles exhibit a higher antibacterial activity versus a gram-negative and gram-positive pathogen [9]. For synthesizes of NiO NPs have been used such as chemical co-precipitation method [10], sol-gel method [11], hydrothermal method [12], micro-wave method [13], reflux method [14], thermo-chemical process [15], electro-deposition [16], electro-spinning method [17], and pulsed laser ablation [18] etc. Among the various approaches, chemical precipitation method is usually adopted for the preparation of nickel oxide nanoparticles because it is simple, low temperatures synthesis, safe and low cost. Even though a few studies have been carried out structural, morphological, magnetic and antibacterial activity on NiO is not insufficient.

In the present work, we complete an effort to study the effect of the, structural analysis, functional analysis, morphology, elemental analysis, and magnetic behavior of the synthesized products of NiO NPS were reported through XRD, FTIR Spectroscopy, SEM/EDX, and VSM.

2. EXPERIMENTAL DETAILS

2.1 **Materials and Methods**

All the chemicals were purchased by Sigma Aldrich AR grade. Aqueous solutions containing stoichiometric ratio of the precursors for Nickel (II) nitrate hexahydrate (AR), Sodium hydroxide (AR) and distilled water were used as precursor materials for the synthesis of NiO NPS.

2.1.1 SYNTHESIS OF NiO NANOPARTICLES (NiO NPS)

The synthesis of NiO NPS at 80^oC was done by adding the Nickel (II) nitrate hexahydrate (0.1M) in distilled water (100 ml) and was wholly dissolved using magnetic stirrer for 10-25 minutes. In another beaker, NaOH pellets (1M) were put in distilled water (100 ml). The required amount of sodium hydroxide (NaOH) solution was added drop by drop wise to the aqueous solution of Nickel (II) nitrate hexahydrate with constant stirring until pH becomes 12. The solution was stirred continuously for 1 hrs at 80^oC. Afterward, slowly a green color precipitate was formed. The green color precipitate was washed 3-4 times with double distilled water, acetone and ethanol. The precipitate was dried at 100^oC for 6h. After that, the material will be in the form of crystal then grinded and calcinations at 600 ^oC and 800 ^oC.

2.2 Characterization of nickel oxide nanoparticles (NiO NPS)

The phase confirmation of the pure NiO NPS was characterized in X-ray diffraction technique using PW3040/60 X'pert PRO PX-ray diffractometer with Cukα radiation (λ = 1.54060 Å) at 40 (kV) and 30 mA. The scans for two theta range from 20° to 80° available at Allagappa University, Karaikudi, and Tamilnadu, India. The chemical composition and functional groups of the NiO nanoparticles were explore by FTIR measurements using a PerkinElmer spectrometer (spectrum-1000). FESEM was well known for utilizing a scanning electron microscope, EVO18 MODEL available at SRM University. The Magnetic behaviors were analyzed in make lakeshore; model: 7410 series at room temperature (RT) available at CIF, IIT Guwahati, (Guwahati).

3. Result and discussions

3.1 Powder X-ray diffraction pattern (structural analysis)

The most useful technique is powder X-ray diffraction (PXRD) is used for the phase identification and the prepared samples were investigated for the structural, purity and crystallinity of the nanocrystals. The PXRD pattern of nickel oxide nanoparticles are shown in fig.1 at as-prepared, 600^oC and 800^oC. It is clearly shown that, the observed peaks at (111), (200), (220), (311) and (222) are without impurity phases. All the peaks are compared with the Joint Committee of Powder

Diffraction Standards Card number 71-1179. The intensive and good sharp peak at (200) indicates that NiO NPS are well crystallized and also have high purity and low particle size. The NiO NPS is found to contain a face centered cubic (FCC) structure with a lattice parameter a=b=c= 4.1780°A. In this result, the author says that the peaks are slightly shifted due to increases of calcinations process.

The average crystallite size of NiO NPS is determined using Debye-Scherer equation [19,20].

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

The micro strain (ε) is determined using the relation:

$$\varepsilon = \frac{\beta \cos\theta}{4}$$

The dislocation density (δ) is calculated using the formula:

$$\delta = \frac{1}{D^2}$$

The lattice parameters (a=b=c) for the cubic phase are calculated by the equation:

$$\mathrm{d} = \frac{\mathrm{a}}{\sqrt{\mathrm{h}^2+\mathrm{k}^2+\mathrm{l}^2}}$$

Where “D” the crystallite size, “λ” the X-ray wavelength (1.54187°A), “K” is a constant and its value is 0.9, “θ” the Braggs diffraction angle, “d” the lattice spacing of the crystal planes (hkl) and “β” Full Width Half Maximum (FWHM) of the diffraction peak. The average crystallite size of nickel oxide nanoparticles increases from 17.89 nm, 32.58 nm and 52.37 nm as the increases calcination temperature. Basically, the crystalline size increases but micro-strain and dislocation density decreases when calcination temperatures increases in NiO NPs. These calculated values are shown in below table1.

Table.1 The structural parameters including crystalline size, dislocation density, micro-strain, lattice parameters and band gap of NiO NPS.

Temperature (°C)	***Average (D) (nm)***	***Micro-strain (ε)***	***Dislocation density(δ)***	***Lattice Parameter(a=b=c)***
As-prepared NiO	17.89	0.010128	0.006946	4.1764
600 °C	32.58	0.006051	0.001022	4.1589
650 °C	52.37	0.000724	0.000497	4.1655

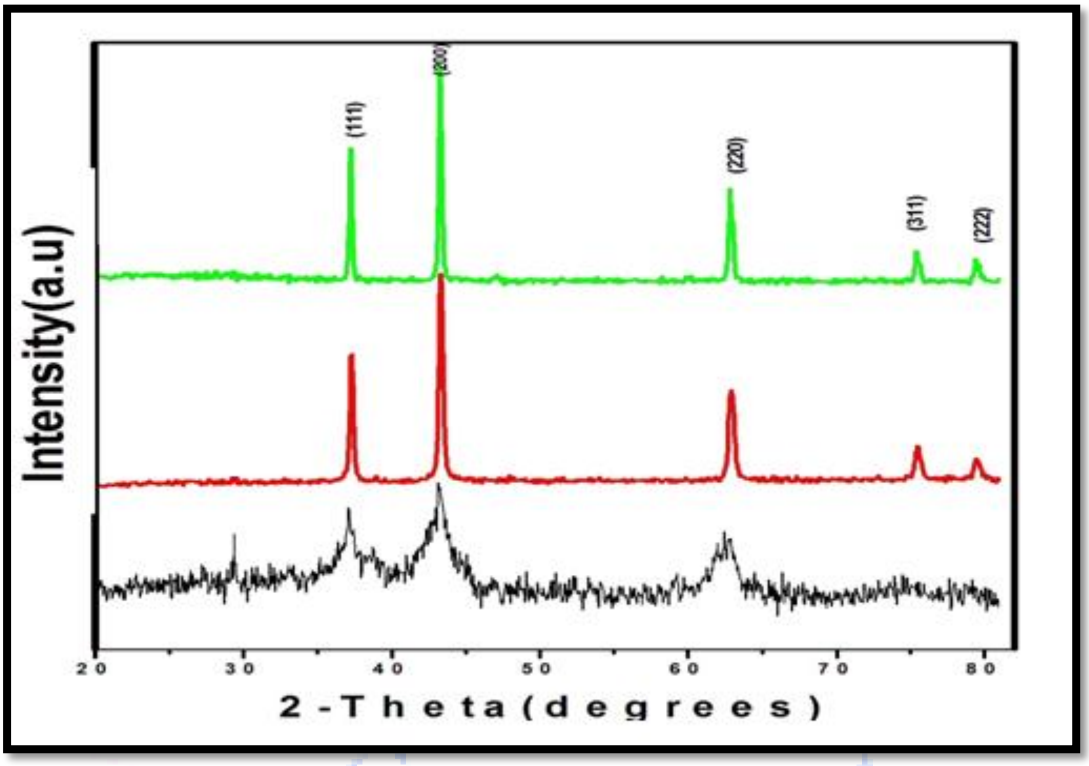

Figure.1. X-ray diffraction patterns of as-prepared and NiO (600 and 800^{o}C) NPS

3.2 FTIR Analysis

The FTIR spectra of as-prepared and two calcinated products obtained at 600 ^{o}C and 800 ^{o}C as shown in fig.2. The wide absorption band occurs around 400-600 cm^{-1}, allotted to the Ni-O stretching vibration mode which confirms the arrangement of NiO NPS and also the broadness of the amalgamation band indicates that the powder of NiO NPs [21]. The broad absorption peak around 3428-42 cm^{-1} is attributed due to the OH-group stretching vibrations [22] and the weak band at 1636 cm^{-1} is due to the H-O-H bending vibrations of irrigating molecules there in band [23]. The peak at 1386 cm^{-1} is corresponding due to the bending vibrations of Co_3^{2-} bond. Additionally, some amalgamation bands appearing in the region of 1150-850 cm^{-1} are due to O-C=O the symmetric stretching and asymmetric stretching mode of vibration molecule in air [24]. This information confirms the occurrence of hydroxyl and carbonyl group in the calcinated powder, which indicates the tendency of NiO powder for physical absorption of water and carbon-dioxide. A similar observation was also reported by Wei et al [25]. At 800^{o}C one more peak occurs at 2926 cm^{-1} due to C-H stretching vibrations. Generally, the metal oxides nanoparticles show absorption bands below 1000 cm^{-1} which arising due to the inter-atomic vibrations. In our result, the two peaks are located at ~ 421 cm^{-1} to 481 cm^{-1} calcinated at 600 ^{o}C and 800^{o}C, related to the Ni-O stretching mode of nickel oxide, which confirms the presence of NiO NPS in the crystalline phase. The peaks are shifted due to the calcination process or due to the crystalline particle size. We can easily understand in table 2 and Fig.2.

Table 2 Functional group analyses of NiO nanoparticles

Reference absorption bands	vibration assignments	Experimental absorption		
		As-prepared	**600 °C**	**800 °C**
400-600 cm^{-1}	Ni-O Stretching	420 cm^{-1}	421cm^{-1}	481cm^{-1}
1434 cm^{-1}	C-O Stretching	1386 cm^{-1}	1384 cm^{-1}	1384 cm^{-1}
1599 cm^{-1}	H-O-H band	1636 cm^{-1}	1633 cm^{-1}	1611 cm^{-1}
2950 cm^{-1}	C-H Stretching	-	-	2926 cm^{-1}
3432 cm^{-1}	O-H Group Stretching	3428 cm^{-1}	3421 cm^{-1}	3424 cm^{-1}

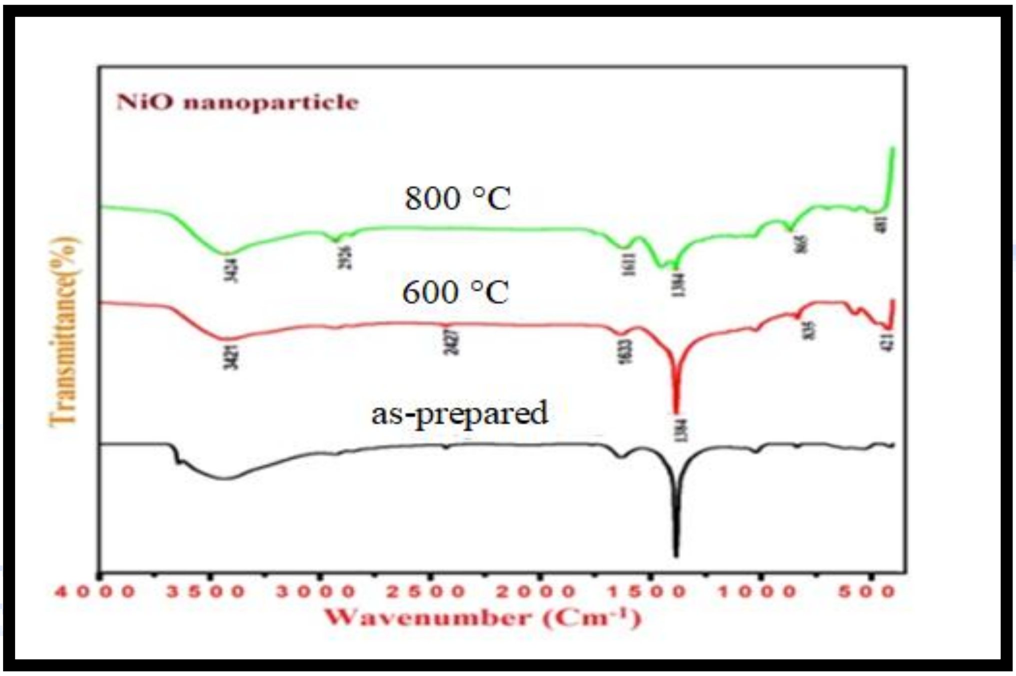

Figure.2. FTIR spectrum of NiO nanoparticles.

3.3 SEM/ EDX Analysis

The surface morphology of the as-prepared and 800°C of NiO NPS have been investigated by using Scanning Electron Microscope. The SEM Nanograph of the as-prepared precursor magnifications is shown in figure 3(a), which shows the typical morphology with spherical coalesced layer structure. After heating at 800°C (with stacked layered structure), changed to subunits of nanoparticles of NiO spherical stacked in nanometer-size particles are shown in fig 3(b). The figure (b) shows the SEM images of NiO NPS at high magnification, which suggest that the NiO NPS is spherical in shape with smooth surface and the crystallite size of nanoparticles [26]. The spherical nano-sizes were successfully done because of the appropriately selected optimum calcination temperature which shows much influence in the particle size of NiO NPS. It is clearly seen that the particle size increases with increasing calcination temperature. The NiO NPS were calcinated at 800°C has spherical shaped nanoparticles. Past reports discovered that agglomerated nanoparticles have higher biological activity

[27]. The elementary composition of NiO NPS is determined by using the energy dispersive X-ray spectroscopy (EDX). In figure 3(c) confirms the occurrence of Ni and O in the synthesis of NiO NPS.

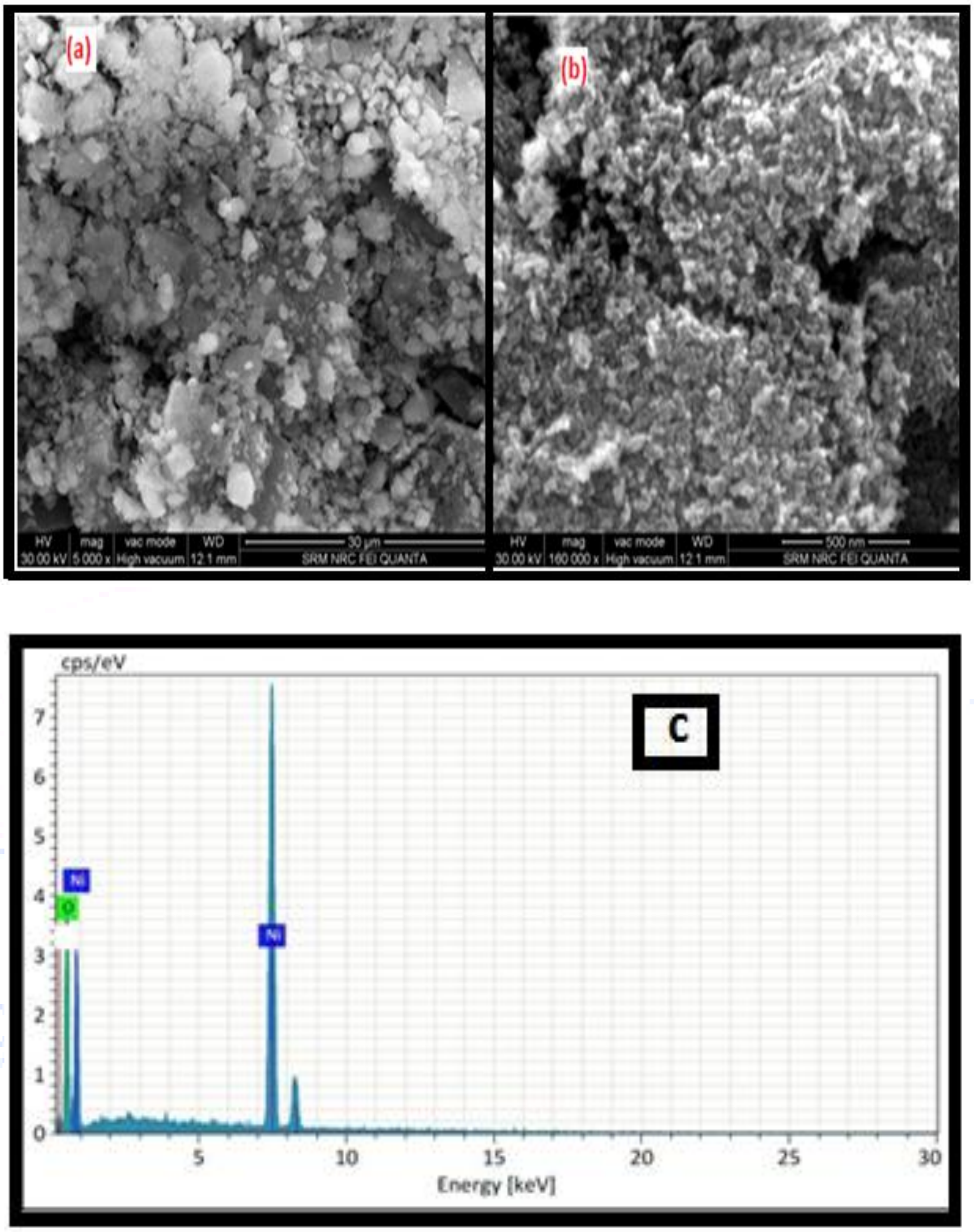

Figure.3 SEM images (a) as-prepared, (b) 800^{o}C of NiO nanoparticles, (c) EDX image of NiO nanoparticles.

3.4 Magnetic properties (VSM)

Figure 4 shows the M-H loops (M vs. H) of nickel oxide nanoparticles at as-prepared, 600°C and 800°C. The retentivity (Mr), coercive field (Hc) and saturation magnetization (Ms) of NiO NPS were originated super-paramagnetic and ferromagnetic in nature [28]. As because of the high coercivity was observed in all NiO NPS due to small particle size, there is a clear evolution of magnetic behavior with increasing calcination temperature with an improved coercivity value. In present work, it is well known that pure NiO NPS at two different temperatures are ferromagnetic in nature. At 60^{o}C temperature which can be related to the uncompensated Ni^{2+} surface spins. This surface spin may be yield as such ferromagnetic behaviors but when it is bulk form it is antiferromagnetic in nature [29]. The crystal structure and particle size plays a leading role for formative magnetic properties like Ms and Hc, which shows that our samples are ferromagnetic behavior. The squareness ratio [SQR = Mr/Ms] is the most important for measurements the best quality of magnetic materials [30]. These results indicate that the coercivity field, retentivity field and saturation magnetization values are increases and decreasing values with increasing calcination. From different magnetization on studies, it is well known that the magnetic properties of NiO have shown different features such as super-paramagnetism, ferromagnetic and antiferromagnetic due to the quantum size effect and other parameters [31].

Table 4. Shows our result, it clearly shows that SQR ratio < 0.8 for all samples that indicates the particles are interact by magneto-static interaction and shows the decreases anisotropy in crystal lattice. It has been found that nickel oxide nanoparticles are synthesized at 60^{o}C temperature shows promising magnetic behavior as compared to others. This type of process materials are easily magnetized and demagnetized. Such materials can be used as soft-magnetic materials for constructing in transformers and cathode material batteries. The soft materials have some more properties, if the hysteresis loop is small so that hysteresis loop loss will be very less. These materials are free from irregularity like strain (or) impurity. The saturation magnetization at 800^{o}C can benefit for mostly transformers, biomedical applications, such as drug delivery, contrast agent materials for the magnetic resonance imaging (MRI) and also bio-sensing applications etc [32]. Therefore, the magnetic properties correlate to structural behavior especially crystallinity, shape, size, strain and so on. This result is in good agreement in previous reports of Ezhilarasi, A. et al [33].

Table3. Magnetic parameters of NiO NPs

Temperature (°C)	**VSM of Hysteresis Parameters**			**SQR=Mr/Ms**
	H_C (Oe)	**M_S (emu/g)**	**M_R×10^{-3}emu/g**	**SQR**
As-prepared	43.24	1.2494	06.1852	0.004
600 °C	181.68	0.2681	13.0050	0.048
800 °C	160.93	0.2361	19.0534	0.080

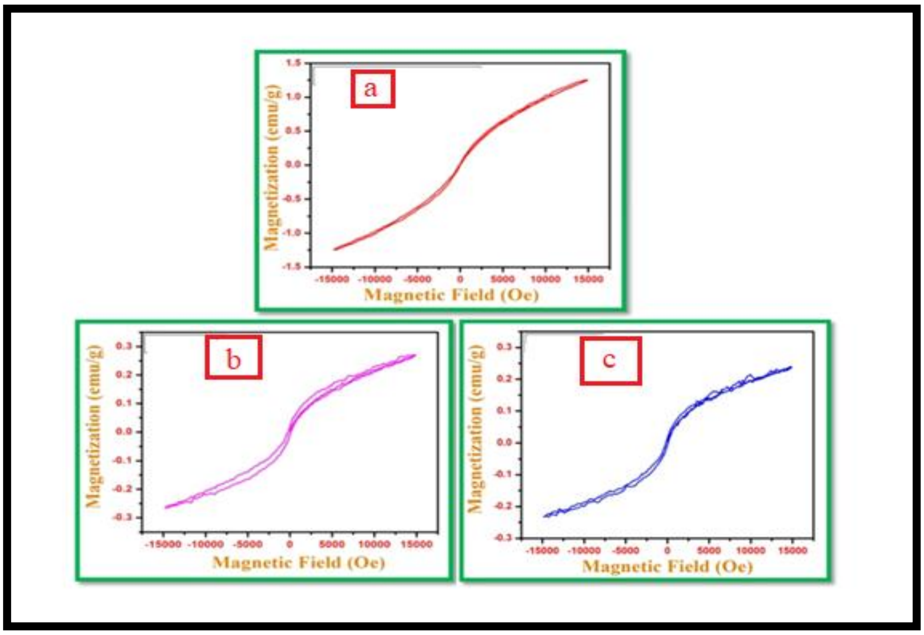

Figure.4 shows M-H curve of NiO nanoparticles (a)As-prepared, (b)600°C and (c)800°C

Conclusions

In this study, nickel oxide nanoparticles were synthesized successfully by precipitation method. XRD pattern affirms polycrystalline nature with FCC structure and crystallite size increases with an increase of calcinations. The FT-IR studies affirmed the Ni-O bonding in the lattice structure of prepared products. The SEM images affirmed that the NiO NPS were spherical particle and high agglomerations were obtained. The magnetic measurements show the weak ferromagnetic behavior for the prepared sample.

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