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## Study the Effect of Doping with Manganese Ions on Some Properties of Nickel Oxide Nanoparticles

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**Abstract** - Nickel oxide nanoparticles impregnated with different molar pence manganese ions were prepared by sol-gel method, using nickel nitrate as a base material, citric acid, white vinegar, sodium hydroxide and manganese nitrate for the doped samples. X-ray diffraction examination confirmed that the harmful substance has a face-centered cubic crystal structure with a lattice constant of 4.18090-4.17697 inxtrum. On the other hand, the FE-SEM (Field Emission Scanning Electron Microscope) assay with the prepared powders within the nanoscale proved the presence of all the elements of the nanocomposite. On the other hand, the Vibrating-sample magnetometer assay proved that the nickel oxide nanocomposite doped with manganese ions has a narrow hysteretic ring and accordingly it can be utilized in transformer cores.

# Keywords: Ni<sub>1-x</sub>Mn<sub>x</sub>O, Sol-Gel, Nanoparticles, Structural Properties, VSM

#### 1. INTRODUCTION

Nickel oxide is a mineral composed of nickel and oxygen NiO, a transition metal oxide having a cubic lattice structure, is an important transition metal oxide. It has gotten a lot of press because of its prospective uses in catalysis [1] gas sensors [2, 3], battery cathodes [4], electrochromic films [5] and magnetic materials [6-8].

Because of its particular features, NiO is one of the metal oxides that is finding its way into the majority of these applications. The FCC structure of NiO, as well as its high band gap of 3.6-4.0 eV, distinguish it [9].Because of their enormous surface areas, peculiar adsorptive characteristics, surface imperfections, and rapid diffusivities, nanosized crystalline metal oxides have piqued researchers' curiosity in recent years. Alkaline batteries, gas sensors, catalysts, in addition, p-type transparent conducting, electrochromic, and magnetic materials.

A few applications for NiO nanoparticles are the cathode and the anode of solid oxide fuel cells [10].

Richardson et al. published the first study of sizedependent magnetic characteristics in NiO. Anomaly magnetic features of antiferromagnetic NiO NPs, such as high moments and coercive forces, according to Kodama et al. (1997), can lead to a novel finite size effect. In which there is a fundamental shift in the magnetic order throughout the particle due to the diminished coordination of surface spins [11]. Chemical precipitation, microwave-assisted methods, anodic arc plasma approach, and sonochemical techniques have all been used to make nanocrystalline NiO powders. Sol-gel has lately developed as a viable method for producing phase-pure oxide nanocrystalline powders [12].

In this paper, we show how to make NiO nanoparticles utilizing an auto combustion Sol-Gel process with nickel nitrate as a precursor. The purpose of this study is to provide a simple approach for manufacturing NiO nanostructure utilizing inexpensive chemicals and simple processing.

#### **EXPERIMENTAL METHOD**

To prepare the NiO nanoparticles by (Sol-gel) technique, the following steps were followed: Take 29.0795 g (by using a sensitive scale with four digit levels of measurement) of aqueous nickel nitrate Ni (NO<sub>3</sub>) 2.6H<sub>2</sub>O dissolved in 100 ml of ionic water. On the other hand, 21.014 g of citric acid  $C_6H_8O_7H_2O$  was added dissolved in 100 ml of ionic water. Then the solutions were mixed together for 30 minutes at RT. The pH function of the solution was adjusted by adding ammonia NH<sub>3</sub> solution until pH -4 was reached. Then the temperature of the solution was raised to 90°C. It was observed that the water had evaporated and while mixing the solution, the solution turned into a viscous gel liquid and later turned into a dry gel. The temperature was increased to 250°C resulting in spontaneous combustion and eventually turning into a fine and fluffy powder, then the heat source was turned off and the material was left to cool. The powdered nickel oxide then placed in the electric oven for three hours at 700°C and then left to cool for 24 hours. Finally, the material was ground to obtain a fine powder of NiO nanoparticles.

#### 2. RESULTS AND DISCUSSION

#### 2.1. X-ray diffraction

X-ray diffraction was used to study the purity of the phase and crystal structure of produced NiO and Ni<sub>0.9</sub>Cu<sub>0.1</sub>O NPs annealed at 700°C. NiO and Ni0.9Cu<sub>0.1</sub>O are the standard XRD spectra illustrated in Figure 1. The diffraction peaks revert to pure NiO nanoparticles, according to XRD patterns. It can be linked to a face-centered cubic structure. Within the range of  $(20^{\circ}-80^{\circ})$ , five distinct peaks of nickel oxide can be seen in the patterns, which are attributed to the planes of (111), (200), (220), (311), and (222) with lattice constants of, respectively. The preferred orientation was (111) on the plane, which matches the typical NiO of (JCPDS 04-0835) excellently [13].From the Bragg positions, the lattice constant for the FCC crystal structure was calculated and the result is in Table 1.



Figure 1. X-ray diffraction of samples

The Scherer relation was used to calculate the average crystallite size of the samples of nickel oxide nanoparticles produced by Sol-Gel method [14, 15]:

 $\mathbf{D} = \mathbf{K} \,\hat{\boldsymbol{\lambda}} / \boldsymbol{\beta} \cos \boldsymbol{\theta} \qquad (1)$ 

1) The surface area increases as the nanoparticle size decreases, and this is a good feature of nanomaterials. From this value, one may infer the characteristics and kind of the material. The formula used to compute surface area is as follows [16]:

 $S_A = 6000/D \rho$  .....(2)

 $S_A$ : The surface area (m<sup>2</sup>/g). (D): particle size (nm). ( $\rho$ ): is the density of the material (g/cm<sup>3</sup>).

The results as shown in Table 1.

2) Based on X-ray diffraction data, one of the key markers by which it is possible to determine whether the sample is monocrystalline or polycrystalline is the crystallization factor. It is determined using the relationship shown below [17, 18]:

 $I_{cry}$ : Crystallization coefficient. D: crystal size from Atomic Force Microscopy (AFM) scan or Scanning Electron Microscopes (SEM).

D <sub>XRD</sub>: crystal size using Scherer equation.

The crystal is monocrystalline if the crystallization coefficient is equal to 1; if it is larger than 1, the material is polycrystalline. [19], the results as shown in Table 1.

Sample	a (Å)	D <sub>SH</sub> (nm)	SA <sub>SH</sub> (m <sup>2</sup> /g)	Icr
1	4.17786	43.6	20.21	1.25
2	4.17697	42.2	20.84	1.45
3	4.17717	36.6	24.07	1.66
4	4.18090	42.1	20.83	1.52

**Table 1.** Lattice constant, crystallite size, SA, and Icr of the<br/>samples

#### 2.2. Magnetic Properties

Heat is released as a result of magnetic hysteresis, with the quantity of energy lost being correlated to the size of the magnetic hysteresis loop. Hysteresis losses will always be an issue in AC transformers because the current is continuously changing direction because the magnetic poles in the core frequently switch direction and production losses.

The relationship between Magnetic Field (Gauss) and Magnetic Moment (emu/gr was plotted using the (VSM) Vibrating-sample magnetometer device. In figure 2 it can note that the samples having the least space for the hysteresis loop means that they consume the least energy and therefore these materials can be used as cores for electric motors and transformers because they have the least magnetic loss [20]. It was observed that the limited size versus surface effects on the magnetic properties of NiO systems, and these investigations demonstrate the strong interaction between the effects of Limited size, surface and interface effects, and defects or oxygen vacancies that renders the magnetic properties of NiO very complex [21]. The results of magnetic properties Hc, Hs and  $\mu$ s for the samples are shown in table 2.





Figure 2. Magnetic Moment Vs Magnetic Field of the samples.

Table 2. Values of magnetic properties (Hc, Hs, and  $\mu$ s) for the samples.

Sample	Hc (Oe)	Hs (Oe)	µs (emu/g)
1	-63	2000	0.6
2	-60	2000	0.35
3	-100	8000	0.085
4	-60	8000	0.23

From observing the FESEM images of the produced powders obtained, we find that they are within the nanoscale range at a

rate ranging between 37.51 - 92.01 nm, and the particles had a spherical or semi-spherical shape as shown in Figure 3. As for the Energy Dispersive X-Ray Analysis (EDX) examination and for samples 1, 2, and 3, they stationed peaks at energies  $K\alpha =$  7.480 keV,  $L\alpha = 0.849$  keV belonging to the Nickel element, 0.525 keV belonging to the oxygen, and 8.046 keV,  $L\alpha = 0.928$  keV belonging to Cu for the Cu doped sample 4. The presence of trace elements Pt and C is due to the properties of the assay device as shown in Figure 4.







2





3

4

Figure 3. FE-SEM images of the samples



1



3



Figure 4. EDX analysis of the samples.

### 3. CONCLUSION



The nickel oxide nanoparticles were prepared by a low-cost spontaneous combustion sol-gel technique. Aqueous nickel nitrate Ni  $(NO_3)_2.6H_2O$  and NaOH were used as precursors. Exrd examination revealed that NiO nanoparticles have a face-centered cubic (FCC) structure with a crystal size of 37.51 - 92.01 nm and a lattice constant of 4.18090 - 4.17697 Å. The magnetic hysteresis of the studied NiO nanoparticles showed that it has magnetic properties, and the highest saturation is 0.35 emu/g. The samples have a narrow hysteretic ring with a smaller area of the that mean that they consume the least amount of energy and therefore can be used as cores for electric motors and transformers because they have less magnetic loss.

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