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Structural and Optical properties of Magnetism Oxide and Nickel doped Magnesium Oxide nanoparticles

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ABSTRACT

In this study, un-doped and Ni doped MgO nanoparticles have been synthesized, through a simple precipitation method. To investigate the effect of Ni-doping on the structure of MgO, the obtained nanoparticles were characterized using scanning electron microscopy (SEM). Plate/slice like morphology was clearly observed in the SEM micrographs. FTIR studies also frequency number 841.06 cm^{-1} appeared in the stretching vibration of metal oxides Mg-O. The irregular nano-sheets that are assembled together by weak interactions provide the plate-like morphology with an increase in unsaturated Ni atoms, oxygen vacancies, and defect sites which render the plate –like MgO more active for catalytic reactions.

Keywords: *Ni doped MgO nanoparticles, Sem analysis, XRD.*

1. INTRODUCTION

The metal elements are able to form a large diversity of oxide compounds. These can adopt a vast number of structural geometries with an electronic structure that can exhibit metallic, semiconductor or insulator character. Even though many reports are available for metal oxide nanoparticles formation (MnO2, Co_3O_4, ZnO, NiO, etc [1-4]. Among different categories of nanoparticles, there is a special interest in transition metals such as Ni, Co, and Fe because of their potential properties and applications in opto-electronics, electronics, sensing, medicine, and catalysis [5]. MgO is an interesting basic oxide that has many applications in catalysis, adsorption and in the synthesis of refractory ceramics. The usage of materials in day-to-day lives of human beings is inevitable in nature and the demand for new materials for different applications like solar cells, energy storage devices, magnetic materials, biosensor, dye degradation effect and so on is ever increasing [6]. MgO is an important material, which used in many applications like catalysis, toxic waste remediation, paint, superconducting products, anti-bacterial activities against food borne pathogens [7-8]. These oxide materials can be prepared by different synthesis methods such as solution combustion [9], Co-precipitation [10], Sol-Gel[11], hydrothermal[12], Solvothermal[13]. These samples were synthesized under standard laboratory conditions in clean room temperature.

2. EXPERIMENTAL PROCEDURE

The procedure of Nickel doped Magnesium Oxide powder was prepared by following way to the precursor was added 50 ml of de- ionized water was stirred to about 3 hours mixed with Magnesium nitrate. Then aqueous NaOH was added drop wise to form the precipitate of magnesium hydroxide. The excess of amount of NaOH was added continuously until the value of pH is approximately 12-14 to ensure completion of precipitation. Then the hydrated hydroxide mixture is transferred to a round bottom flask fitted with a water condenser and placed in a rotomandle. The mixture was continuously stirred for five hours and temperature was maintained at 80 to 100°C, after continuous string crystalline powder was found under the bottom of the flask and the powder was washed several time by the de ionized water to remove the remaining the highly ionic particle. Washed sample powder was filtered by the Waatman filter paper. Hence the power was dried at 100°Cwith the help of in the oven, then powder was grained into pure powder of MgO and it was calcined 400°C, all ratio of nickel (1-5%) doped MgO was prepared by same procedure and it was analyzed with different characterization.

3. RESULTS AND DISCUSSION

3.1. XRD Interpretation

XRD was used to characterize pure and 1%, 2%, 3%, 4% and 5% of the Nickel doped Magnesium Oxide Nano powders. The sample was scanned in the range of10º to 80º for a period of 5 sec step of scan mode. The diffraction pattern presence to peak corresponds to the reflection plane of the monoclinic structure of the metallic Magnesium Oxide and Nickel. The small fraction of identified

phase of α Magnesium Oxide was observed from the JCPDS file (2013). The average crystalline size has been determined by Debye Scherrer formula,

$$D = \frac{0.94\lambda}{2\beta\cos\theta}$$

Where, λ - the wavelength of the X - ray, β – the full width half maxima, θ – the diffraction angle.

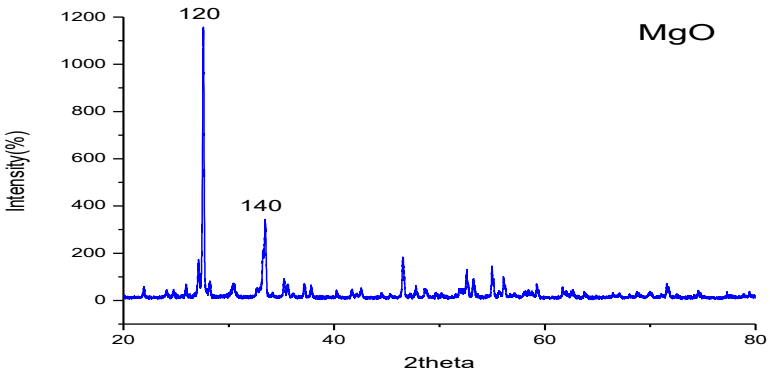

Fig.1. XRD patterns of pure MgO

The calculated lattice strain from the observations is 0.0018, 0.0013, 0.0012, 0.0010, and 0.0013. The comparative table indicate that the XRD patterns of the Nickel doped Magnesium Oxide corresponding to the Monoclinic structure PDF card no: 71-0465 primitive lattice of space group is P_{21} / C(14) and lattice parameters a = 5.849, b = 8.164, c = 7.510, α = 90º, β = 112.97º, γ = 90º. The α - phase characteristic peak appears when the temperature was increased from the room temperature 400ºC.

The major peaks of the XRD pattern of pure and Nickel doped Magnesium Oxide at 2θ are 26.9458, 27.392, 28.1174, 26.966 and 28.0688 corresponding to the diffraction of the miller indices ($\bar{1}12$), (120), (012), ($\bar{1}12$) and (012).The grain size of Nickel doped Magnesium Oxide vary from 17.4 to 82.1 nm for pure and 15.2 to 64.03nm, 12.1 nm to 79.7 nm, 13.6nm to 54.8nm and 23.1nm to 75.7 nm for 1%, 2%, 3%, 4% and 5%and the average particle size varies from 16.28 to 71.26nm respectively[14].

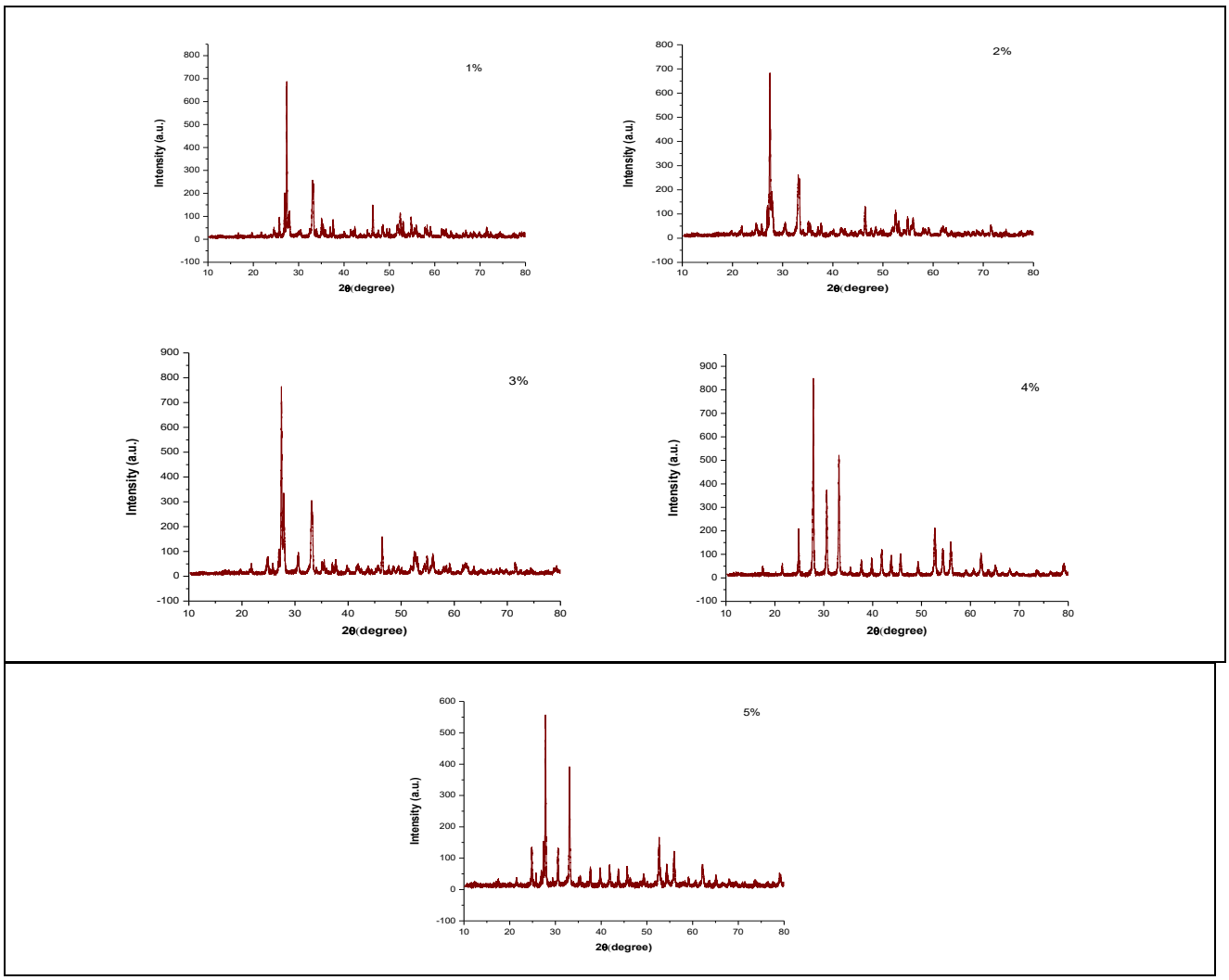

Fig.2. XRD Pattern of Nickel (1%,2%,3%,4%&5%) doped Magnesium oxide

3.2. FTIR Interpretation

FTIR spectra analysis of the pure and nickel doped Magnesium Oxide of different ratio of a percent 1%,2%, 3%,4%&5% the structure of the component was identified from the monitor spectra which is annealed by the Berkin Elmeir. From the spectrum follows that the unknown phase is probably Magnesiumhydroxide, which can be transformed into single phase MgO by annealed 400ºC. The water bond as O-H stretching mode in the two peaks around 1600 cm^{-1} and 3600cm-1 the main influence on the composition of reaction products was that of pH>14 of reaction medium [14].The crystalline structure also confirmed by the range of 840cm^{-1} as Mg-O metaloxide confirmed that the structure of monoclinic. From the table shows that the varies vibration assignment the intensity distribution of the functional group.

Table 1. FTIR parameters of Ni doped MgO Nanopowders

S. No	Observed Wavenumber (cm^{-1})	Standard Wavenumber (cm^{-1})	Vibrational stretching	Intensity	Component groups
1	2373.52	2350	CO_2	Strong	Carboxyl
2	2346.33	2350	CO_2	Strong	Carboxyl
3	1460.32	1400	NO_2	Weak	Amine
4	1383.56	1400	MgO	Weak	Amine
5	850.99	840	MgO	Strong	Metal oxide
6	508.75	400-600	MgO	Strong	Metal oxide

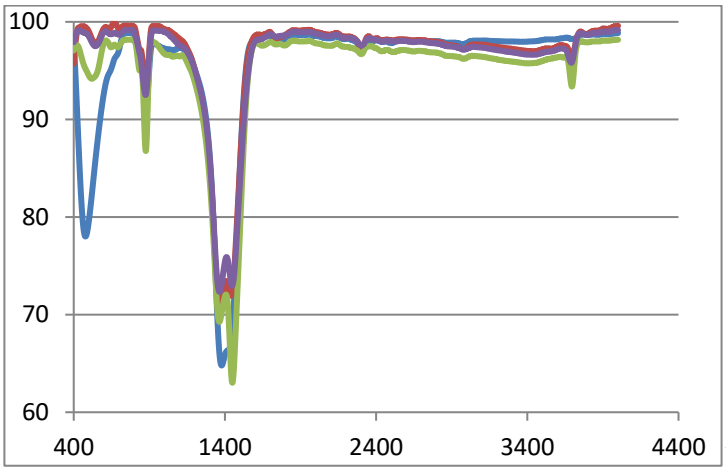

Fig.3. FTIR interpretation of Nickel (1%, 2%, 3%, 4% and 5%) doped Magnesium Oxide

3.3. UV-DRS -Studies

UV diffuse reflectance analysis of pure and Ni doped powder was determined by (UV-2102 PCS spectro photometer).the UV visible diffuse reflectance spectra of as prepared pure and Ni-MgO sample was annealed at 400°C at different ratio for 6 hr. The bandgap absorption edges figure (3&4) determined to the 524.64 nm,532.11 nm,538.07 nm and 539.80nm .Which is corresponding to the bandgap energy of 2.3085 eV,2.3344eV,2.3676 eV and 2.3011 eV respectively.

The significant increases in the absorption wavelength shorter than about 539.80 nm could be assigned to the intrinsic band gap absorption of Ni doped MgO with increasing the percentage of concentration of Ni - MgO. The obtained sample shows a stronger absorption in UV- visible range and blue shift transition in the bandgap transition. The blueshift could be ascribed to the increasing of crystal particle size.

From the figure (3&4) the absorption edges of the crystalline powders obtained by co-precipitation method shows a marked blue shift to red shift as compared to that of the sample obtained by the solid state reaction. The result may describe to the quantum size effect of the nanosized Ni-MgO particles obtained by co-precipitation method which is consistent with the results of XRD and SEM.

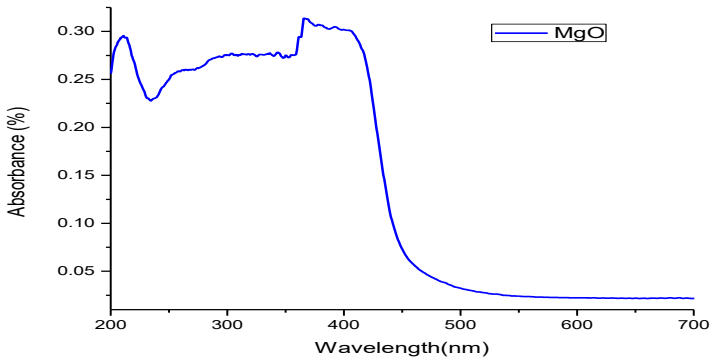

Fig.3. UV DRS Absorbance spectra of pure MgO

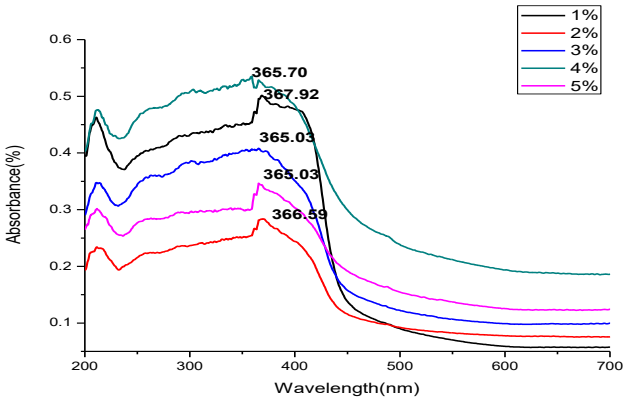

Fig.4. UV DRS Absorbance spectra of Nickel (1%, 2%, 3%, 4% and 5%) MagnesiumOxide

3.4. SEM analysis of Ni doped MgO nanopowders

Figure.5 shows the morphology of the samples observed by SEM. Plate /slice like morphology is clearly distinguishable in the pictures. Some clustered particles are also found especially in Ni doped MgO nanoparticles. The surface area of Ni nanoparticles doped by MgO appears to be greater in these pictures due to the finer morphology of the plate-like features. The plate/slice -like MgO benefits from a larger surface area, bimodal pore size distribution, higher reducibility, and superior catalytic activity. The irregular nano-sheets that are assembled together by weak interactions provide the plate-like morphology with an increase in unsaturated Ni atoms, oxygen vacancies, and defect sites which render the plate –like MgO more active for catalytic reactions.

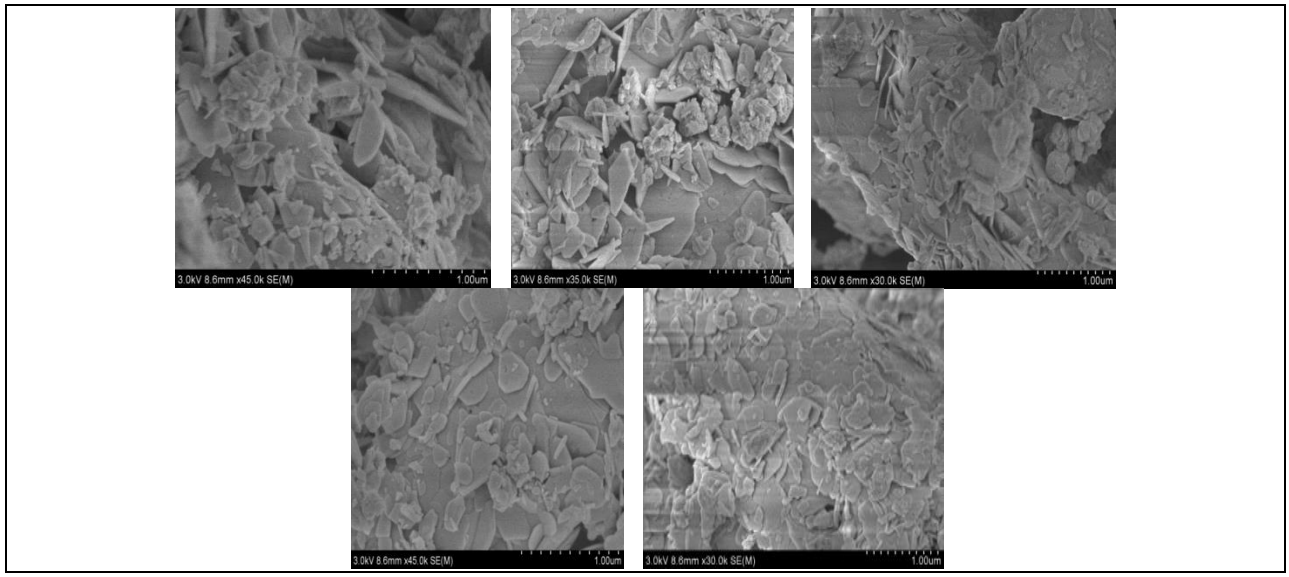

Fig.5. SEM image of Nickel doped Magnesium Oxide.

3.5. EDX-Studies on nano materials

Figure 6. Shows the EDX-spectrum of synthesis of pure and Ni doped MgO structure of nanosheets revealed as atomic ratio 76.18 and 23.82. That is nanosheets were Mg and O elements are rich. Then fig6 represents the Ni and MgOatomic ratio of 84.92ofOxygen and 15.08 of Magnesium (Mg) very less percentage of Nickel (Ni) shows in figure.

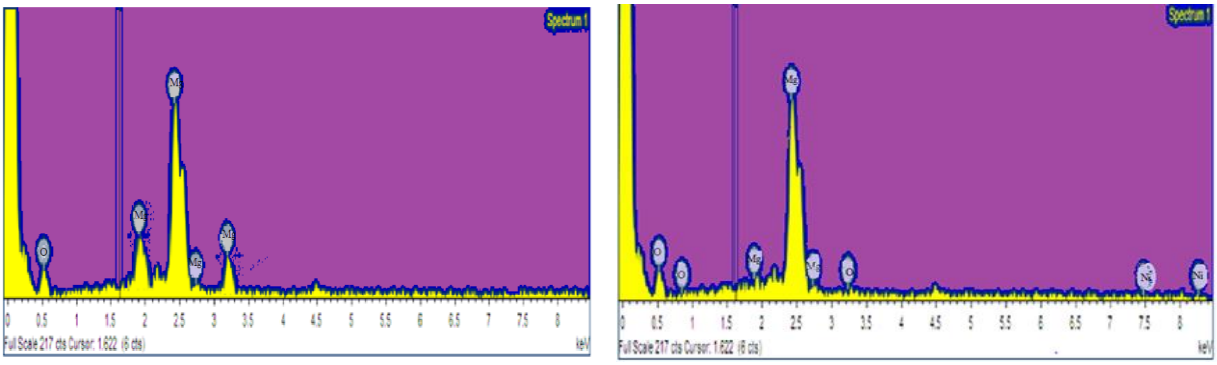

Fig.6. EDX spectrum of MgOand 1% Ni doped MgONano sheets.

4. CONCLUSION

A co-precipitation procedure was used to prepare the pure and Ni doped Magnesium Oxide (MgO) annealed at 400°C. Optical absorption band gap energy of the Ni doped MgO is 0.03eV which smaller than that pure MgO from the observation of UV-DRS.

Pure MgO and Ni - doped MgO crystalline material as MgO system was confirmed by the XRD pattern with SEM images clearly shows that the synthesis sample consist of Nano plate- like architecture in large quantities. It is confirmed that the average grain size of the particle size varying from 16.28 nm to 71.26 nm scale of different percentage of deponent of Ni. This is two possible explanations for the low temperature co-precipitation method. FTIR studies also frequency number 841.06 cm^{-1} appeared in the stretching vibration of metal oxides Mg-O. The irregular nano-sheets that are assembled together by weak interactions provide the plate-like morphology with an increase in unsaturated Ni atoms, oxygen vacancies, and defect sites which render the plate –like MgO more active for catalytic reactions.

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