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## SOL-GEL SYNTHESIS AND CHARACTERIZATION OF NIO NANOPARTICLES

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**ABSTRACT:** In order to study the roles of the crystallite size of an active phase in a catalytic reaction it is of utmost importance to be able to synthesize pure phases of crystallite in the desired size range with a narrow size distribution. The method to produce a nano of Nickel oxide ( $NiO$ ) is described. Nanoparticles (NPs) of  $NiO$  are prepared by thermal decomposition of freshly prepared nickel hydroxide by a sol gel route at  $300^{\circ}C$ . This sample is characterized by X-ray diffractometer (XRD) and UV-visible spectroscopy. The average crystalline space or the separation between atomic planes of ( $NiO$ ) NPs is found to be about (2 - 5nm) and the average of particle size is (9- 19 nm). According to our X-ray and neutron diffraction data, all NiO powders are antiferromagnetically ordered at room temperature.

المستخلص- إن التفاعل المحفز في دراسة الجسيمات النانوية (في حجم النانو) ذات الطور النشط ذو أهمية عظمى لتحضير أطوار نقية من البلورة (المعدن الدقيق) في مدى حجمي منشود في توزيع حجمي ضيق. وفي هذه الدراسة قد وصفت الطريقة التي حضرت بها نانويات أكسيد النيكل وهي طريقة التفكك الحراري لهيدروكسيد النيكل المعد بطريقة الصول جل عند درجة حرارة 300 م. والعينات المحضرة شخست بأجهزة حيود الأشعة السينية ومجهر الأشعة فوق البنفسجية ووجد أن متوسط الفراغ البلوري (البعد الفاصل بين المستويات الذرية) لنانويات أكسيد النيكل (2- 5 نانومتر) ومتوسط حجم الجسيمات النانوية (9-19 نانومتر). وبالرجوع إلى تشخيصات جهاز حيود الأشعة السينية وجد أن كل نانويات أكسيد النيكل ذات مغناطيسية معاكسة.

**Keywords:** NPs; XRD; UV; ferromagnetism

### INTRODUCTION

The object of study of nano ferromagnetism is science, engineering, measuring, modeling and nanotechnology involving objects of dimensions usually in the range from 1 nano meter ( $1\text{ nm} = 10^{-9}\text{m}$ ) to 100 nanometers, and control of matters at the realm of 1 to 100 nanometers where unique <sup>[1]</sup>. This is the range of sizes of many molecule and viruses and is also the characteristic length scale of many physical processes. The lateral dimensions of present day integrated circuit components. As well the dimensions of grains in magnetic recording film media, are contained in this range. Nanoscale is a

magical point on the dimensional scale. Structures in nanoscale (called nanostructures) are considered at the borderline of the smallest of human-made devices and the largest molecules of living systems, moreover, nanotechnology become the main tools of the modern science for this century and future. Our ability to control and manipulate nanostructures will make it possible to exploit new physical, biological and chemical properties of systems that are intermediate in size, between single atoms, molecules and bulk materials <sup>[2]</sup>. Nanomagnetism is the area of research in Nanoscience that deals with the

magnetic properties of objects that have at least one dimension in the nanoscopic range. Nanomagnetism includes in its scope the study of properties and applications of the magnetism of isolated of NPs, nanodots, nanowires, thin films and multilayer's, and also macroscopic samples that contain nanoscopic particles. Materials that contain particles, film and other structures in the nanometric scale are often described as nano structured materials and becomes superparamagnetism materials, some of it is denoted by nanoferroxide ( $NiO$ ,  $Fe_2O_3$ ) materials and are considerable in this search and has similar magnetic, electric, and chemical properties<sup>[3]</sup>. Over the last 10 years, synthesis of  $NiO$  NPs has received significant attention owing to the immense potential of these materials in a broad range of applications which is due to their unique properties including magnetic, catalytic, and electronic properties. The synthesis methods for NPs include well-established processes such as wet chemistry, chemical vapor deposition, physical vapor deposition, ion sputtering, pyrolysis, sol-gel, or flame synthesis<sup>[4]</sup>. Also the  $NiO$  NPs sinters are used in the manufacture of alloy steels and stainless steels.<sup>[5]</sup>

The previous works of  $NiO$  NPs Synthesis and characterization of  $NiO$  NPs (Sakshi Gupta July 2011) prepared  $NiO$  nanoparticles, structure characterization, thermal characterization, and magnetization measurement. Structure and magnetic properties of Nickel Oxide NPs (NN. Mironova – Ulmane) Synthesis and magnetic properties of  $NiO$  NPs. Nickel and Nickel compounds studies by IARC Working Group in 1972, 1975, 1979, 1982, 1987, and 1989 to produced Black  $NiO$  is used in the manufacture of nickel salts, specialty ceramics, and nickel catalysts. Structure and Optical properties of Ni (1-x) Zn<sub>x</sub> O which synthesis by Chemical Spray Pyrolysis Method (CSP) investigated by Rodena siddig University of Dealy. (Sohaib Z. Khan) Characterization of such NPs in terms of size distribution, shape, chemical composition, and phase structure was carried out by transmission electron microscopy (TEM), high-resolution

TEM equipped with energy-dispersive X-ray (EDX), and X-ray diffraction (XRD).

The main Objectives of this work were to Sol – Gel synthesis of  $NiO$  NPs, and to Structural and optical properties of  $NiO$  NPs.

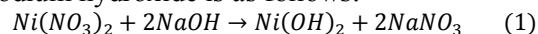
#### **MATERIALS AND METHOD:**

##### **Materials:**

To prepare the supported Nickel Oxide in nano size the following several physical and chemical methods were used during the experimental work. Some materials has been used in the work (nickel nitrate  $i(NO_3)_2 \cdot 6H_2O$ ), sodium hydroxide ( $NaOH$ ). Some interaction of solutions used to decrease the crystallite size a higher number of surface atoms per gram are exposed and available to transfer the electric and magnetic properties<sup>[6]</sup>.

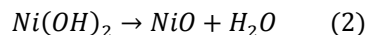
##### **Synthesis of Nickel Oxide Nanoparticles:**

The Nanosized ( $NiO$ ) powders have been produced by several chemical methods. In the present work nanoparticles of  $NiO$  are synthesized by a sol gel method. This process involves the formation of a colloidal suspension (sol) and gelation of the sol to the form a network in a continuances liquid phase (gel). The starting materials are processed to form a sol in water dilute acid. Removal of the liquid from the sol yields the gel. Nano particles of  $NiO$  are prepared decomposition of freshly prepared  $Ni(H)_2$  and the ( $NiOH$ ) is prepared by reacting aqueous solution of 0.1 M nickel nitrate  $Ni(NO_3)_2 \cdot 6H_2O$  and 0.5M sodium hydroxide ( $NaOH$ ). For this  $NaOH$  solution is added drop wise with constant stirring until the PH (( potential of hydrogen) is a numeric scale used to specify the acidity or basicity of an aqueous solution) of the system reaches to 12. The chemical reaction between nickel nitrate and sodium hydroxide is as follows:



In this work,  $NiO$  NPs is prepared by heating the  $Ni(OH)_2$  in air for 3 hours at 300°C

The resulting green gel is washed several times with distilled water. Finally the gel is dried by heating at 100°C for 10 hours. Nickel hydroxide decomposes into nickel oxide on heating as follows:



In this work, the experimental has been repeated four times altering the ratio of solutions and the nanoparticles of *NiO* samples use to prepare particles about 9- 19nm. A detailed characterization of the samples was carried out using XRD and UV – visible spectroscopy. XRD patterns were obtained using Bruker D8 advanced diffractometer using *CuKα* monochromatic radiation ( $\lambda = 1.5418 \text{ \AA}$ ) at room temperature operated at voltage 50 *kv* and current of 40*mA* [7; 8]. The intensity of the diffracted X-Ray beam is recorded as a function of the angle  $2\theta$ . Optical absorption spectra were recorded with the conventional beam of some methods using UV-VIS spectrometer.

### RESULTS AND DISCUSSION:

To study the optical properties (reflectance, absorption, energy band gap and optical conductivity) and the effect of *NiO* UV – visible absorption spectra were recorded in the incident photon wavelength of 400 *nm* to 600 *nm* in the visible light region [9; 10].

According to UV data of Figure 1 if the ratio of the nickel nitrate *Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O* and sodium hydroxide (*NaOH*) for all samples increased (from 1:1 to 1:4) the Reflectance (R), will be decreased. Further analysis showed that the composition remained consistent and also the variation of the size and shape of the NPs did affect their composition, the Reflectance will be calculated by equation (3) [11; 12]. Where *k* is extinction coefficient and *n* is a refractive index.

$$n = \frac{(1 + R)}{(1 - R)} + \sqrt{\left(\frac{4R}{(1 - R)^2} - K^2\right)} \quad (3)$$

In Figure (2) we noted that if the ratios of the sodium hydroxide (*NaOH*) and nickel nitrate *Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O* for all samples increased (from 1:1 to 1:4) the Absorption (A) will be increased, and the equation (4) is calculated the absorption: [13; 14].

$$A = \frac{(1 - R)^2}{2R} \quad (4)$$

And also show in Figure (2) the ratio of the absorbance at the surface plasma resonance peak (*Aspr*) to the absorbance at 450 – 550 *nm* (*A*) in dependence of the logarithm of the particle diameter in the size range from 5 to 80 *nm*. The theoretical data (circles) exhibit an excellent linearity (Figure (2)), and hence the ratio *Aspr/A* should be particularly suitable to calculate the particle diameter (in nanometers) from

$$d = \exp\left(B_1 \frac{A_{SPR}}{A} - B_2\right) \quad (5)$$

Where *d* is a nanoparticle size (the particle diameter in nanometers), *B<sub>1</sub>* is the inverse of the slope (*m*) of the linear fit in Figure 2 and *B<sub>2</sub>* = *B<sub>0</sub>/m* where *B<sub>0</sub>* is the intercept.

In Figure (3) shows the plot line for determining the band gap energy of NPs, the band gap energy of the *NiO* in the range (2.55 *ev*) [15]. If the ratio of the nickel nitrate *Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O* and sodium hydroxide (*NaOH*) for all samples increased (from 1:1 to 1:4) the band gap energy will be increased. The appearance of such larger band gap energy is expected in *NiONPs* because of their small size. This observation is consistence with the previously reported large band gap energy in *NiONPs* and nano-rods. The energy band gaps at the following equation:

$$\alpha h\nu = B(h\nu - E_g)^r \quad (6)$$

Where *E<sub>g</sub>* the energy gap, constant *B* is different for different transitions, (*hν*) is the energy of a photon and *r* is an index which assumes the values 1/2, 3/2, 2 and 3 depending on the nature of the electronic transition responsible for the reflection. The absorption coefficient,  $\alpha$  is given by [11; 16].

$$\alpha = \frac{2.303A}{d} \quad (7)$$

Where *A* the absorption and *d* the particle size. The phase analyses of the samples were performed by X-ray diffraction (XRD) -6000 with *Cu Kα* radiation ( $\lambda = 1.5418 \text{ \AA}$ ) [17; 18]. The XRD pattern of the prepared green colored powder at room temperature is shown in Figure (4) from this pattern, it is clear that the green colored sample is *NiO*, it is also noticed that the

peaks are broadened. This indicated that the sample is Nano crystalline. The average grains sizes, is calculated by using the modified Scherrer formula [7; 19].

$$d = \frac{0.9\lambda}{\cos \theta (B_m^2 - B_s^2)^{1/2}} \quad (8)$$

Where  $\lambda$  is the wavelength of the X-ray used,  $\theta$  is the Bragg angle  $B_m$  is the full width at half – maximum (FWHM) of a peak in radians and  $B_s$  is the FWHM of the same peak of a standard sample. Three most intense peaks are used to calculate the average grains sized by using the modified Scherrer formula. This turns out to about  $3.2\text{nm}$ . The use of  $(B_m^2 - B_s^2)^{1/2}$  instead of  $B_m$  in the Scherrer formula takes care of instrumental broadening. To explain how the XRD characterized the magnetic of materials: The rich polarization dependence of magnetic scattering can be used to extract the magnetic ordering of a material. Circularly polarized (CP) radiation can also be useful in studies of Antiferromagnetic (AF) materials. CP X-rays were used for real-space imaging of chiral domains by helicity-dependent Bragg scattering from the spiral AF state at room temperature.

#### CONCLUSION:

In this work *Nio* nanoparticles are synthesized by thermal decomposition of freshly prepared nickel hydroxide by a sol gel route at  $300^\circ\text{C}$  in air. This sample is characterized by X-ray diffractometer (XRD) and UV – visible spectroscopy. The average crystallite space ( $d$  is the separation between atomic planes) of (*NiO*) is found to be about (2 - 5nm) and the structural parameters, determined by the Rietveld analysis procedure, indicate an expansion of the lattice volume when the average of particle size is (9-19 nm). Our diffraction data indicate that at room temperature all NiO powders are antiferromagnetically ordered and have rhombohedral ( $R\text{-}3m$ ) and cubic crystal phase structures which were single crystal in nature irrespective of the size and shape.

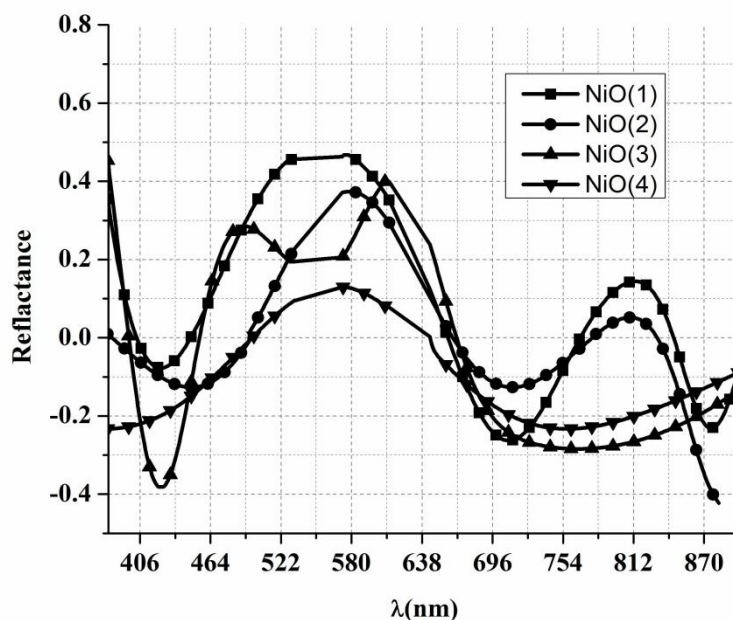
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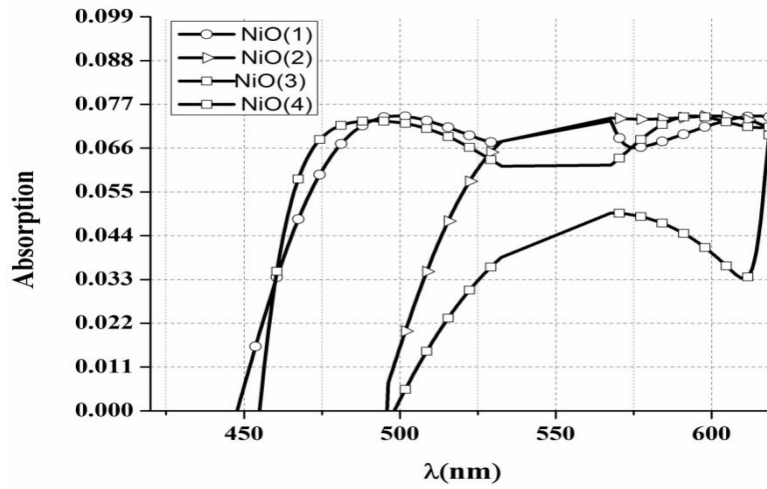
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**Table (1):** The different NPs sizes produce for different ratios of the sodium hydroxide (*NaOH*) and nickel nitrate *Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O*) for all samples.

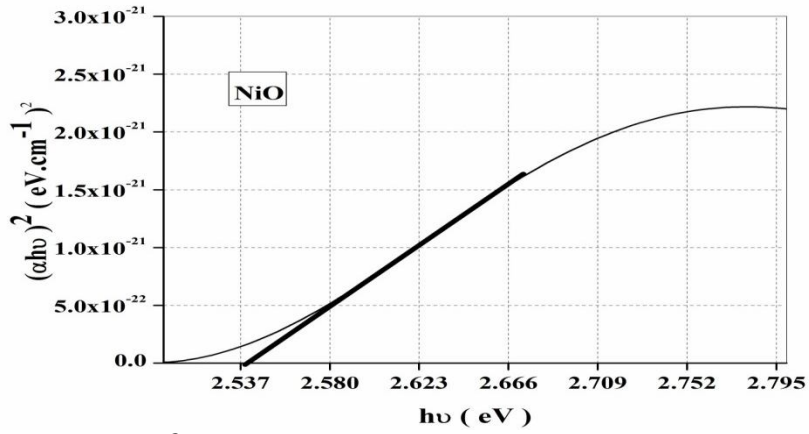
No	<i>NaOH</i> (ml)	<i>Ni(NO<sub>3</sub>)<sub>2</sub></i> (ml)	size(nm)	<i>NiO</i>
1	50	50	16.4	<i>NiO</i> (1)
2	50	100	10.9	<i>NiO</i> (2)
3	50	150	9.8	<i>NiO</i> (3)
4	50	200	18.5	<i>NiO</i> (4)



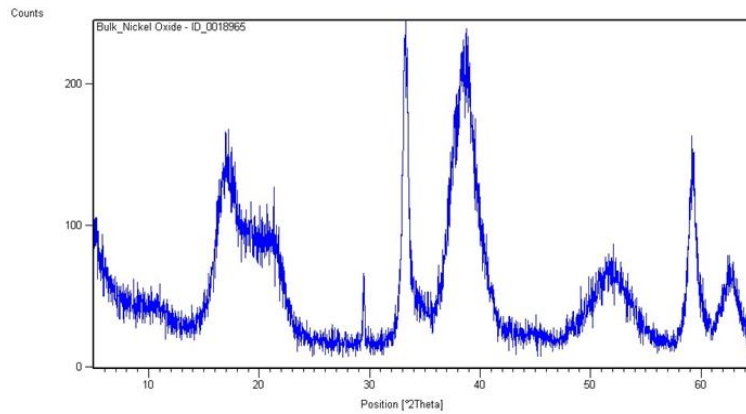
**Figure 1:** The Optical Reflectance spectral as a function of wavelength with different sizes of *NiO* NPs samples.



**Figure 2:** The Optical Absorption spectral as a function of wavelength with different sizes of *NiO* NPs samples.



**Figure 3:** Plot  $(\alpha h\nu)^2$  as a function to photon energy of the *NiO* nanoparticles powder.



**Figure 4:** XRD pattern for *NiO* powders at room temperature.