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Exploring Characteristics of NiO Nanoparticles using Sol-Gel Method

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ABSTRACT

In this article, production of nioxide nanopaticles and its lattice parameters are reported using sol-gel method. The structural & crystallite size studies were performed by utilizing X-ray diffraction. The identification of cubic structure of NiO nanocrystallite is ascertained. X-ray diffraction (XRD) results indicates that the crystallite size is in 20-30nm range with average size of 25nm, lattice parameter a= 4.175A^0 for cubic structure of NiO. Here NiO nanoparticles were annealed at temperature 700˚C.

Keywords: nanoparticles, Precursor, zinc oxide, Sol-gel , X R D

1. Intoduction:

Nickel oxide is a chemical compound with the formula NiO. It is the principal oxide of nickel. It is classified as a basic metal oxide. Several million kilograms are produced annually of varying quality, mainly as an intermediate in the production of nickel alloys. The mineralogical form of NiO, bunsenite, is very rare. Other nickel oxides have been claimed, for example: Nickel (III) oxide (Ni_2O_3) and NiO_2, but they have yet to be proven by X-ray crystallography in bulk. Nickel (III) oxide nanoparticles have recently (2015) been characterized using powder X-ray diffraction and electron microscopy. It is an interesting material because of its chemical stability as well as optical electrical and magnetic properties. Its nanoparticles are used in electro chromic devices smart windows, optical fibres, gas sensors solar thermal absorbers, batteries, transparent conducting layers. Numerous techniques, such as chemical precipitation, magnetron sputtering, and sol-gel have been used to fabricate NiO nanoparticles. Among different techniques for controlled synthesis, sol-gel technique was used to synthesize crystalline and impurity free NiO nanoparticles.The nickel oxide has a history that dates back to 1899, when it was described in a German patent by '**Michalowski'**.[1-20]

2. Experimental work

0.2 M of nickel acetate dehydrate, reducing agent 0.2 M of NaOH were weighted using a weighting balance. Then 50 ml of distilled water was measured by measuring cylinder for each sample. After that, all the weighting samples were dissolved separately in 50ml of distilled water. All the solutions were stirred with constant stirring for about 30 minutes. Then 0.2M of NaOH solution as reducing agent poured to the solution containing nickel acetate with constant stirring by magnetic stirrer for about one hour. Then burette was filled with 100ml of PVA (polyvinyl alcohol) and titrate drop wise to the solution containing reducing agent nickel acetate. After the constant stirring, precipitates were formed. Dried precipitate was calcinated at about 700˚C for 24 hours in muffle furnace. So we have produced nickel oxide nanoparticles. This procedure adopted is "sol gel method". [21-27]

3. RESULT AND DISCUSSION:

XRD-STUDY:

The NiO nanoparticle was performed using CuK_a radiation source of a wavelength $\lambda = 1.54060\text{Å}$ and the diffraction pattern by studied by changing diffraction angle in the range of 35˚ to 90˚. The XRD pattern of NiO nanoparticles synthesised by sol gel technique shows maximum intensity of 18000, which indicate large production of crystallite size (fig.1). The XRD patterns confirm that the formed material is nickel oxide. XRD pattern clearly show the diffraction peaks of the (111), (200), (220), (311) (222) planes.

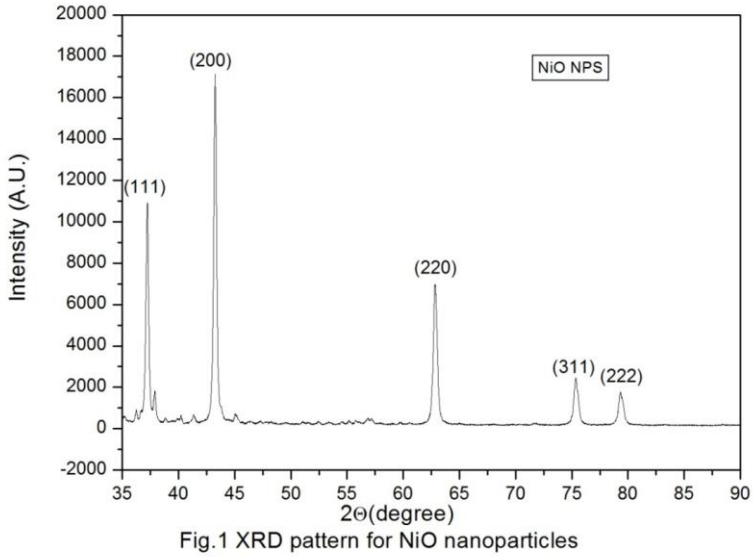


Fig.1 XRD pattern for NiO nanoparticles

The XRD pattern, calculated d-value and lattice parameter confirm that the nanoparticles of NiO has cubic structure. It canbe observed that by increasing the annealing intensity upto 18000 appreciably indicates that growth in the crystalline size of NiO has taken place. The average crystalline sizes D of nickel oxide nanoparticles were calculated by Debye-Scherer equation using full width at half maximum:

$$D=K\lambda/\beta\cos\theta$$

Where D is the crystalline size, β is the broadening of the diffraction line measured at its maximum intensity (FWHM), K is the Scherer constant of the order of 0.89 related to crystalline shape and Cu Kα radiation (λ=1.5406A°)..

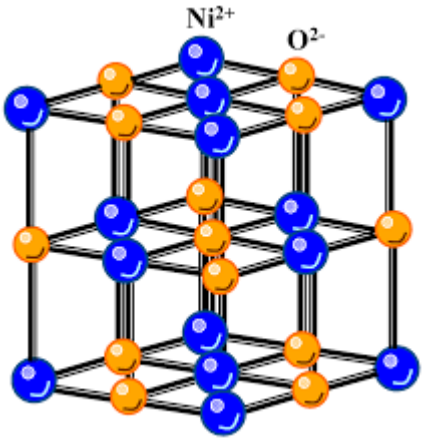
Table1: Crystalline Size(D) of NiO NPs

Angle (2θ)	θ	θ (Radian)	Cos(θ)	β	β (Radian)	βCos(θ)	D	D (nm)	hkl
37.21	18.61	0.324719	0.948	0.271	0.004732	0.00449	305.6	30.559	[111]
43.25	21.63	0.377427	0.93	0.3	0.005232	0.00486	281.8	28.178	[200]
62.84	31.42	0.548382	0.853	0.409	0.007131	0.00609	225.2	22.523	[220]
75.38	37.69	0.657815	0.791	0.457	0.007969	0.00631	217.3	21.734	[311]
79.37	39.69	0.692634	0.77	0.499	0.008703	0.0067	204.6	20.465	[222]
						Avg. (D)=		**25nm**	

The crystalline size 'D' is 25nm

LATTICE STRUCTURE

Nickel oxide NiO is an important transition metal oxide with **Cubic lattice structure.**



a = b = c

Fig.2

For a cubic crystal system the X-ray diffraction patter recorded using CuK_asource (λ=1.54Å) shows a peak at 43.25˚ for (200) plane.

$$\frac{1}{d^2} = \frac{h^2+k^2+l^2}{a^2} \quad \text{---------- (1)}$$

For plane (200), here h = 2 k = 0 l = 0

Put these values in above formula:

$$\frac{1}{d^2} = \frac{(2)^2+(0)^2+(0)^2}{a^2} \quad \text{---------- (2)}$$

According to Bragg's law concept – Bragg's law relate the angle θ to the wavelength of X-rays and the interlayer distance 'd' between the planes of atoms /ions /molecules in the lattice.

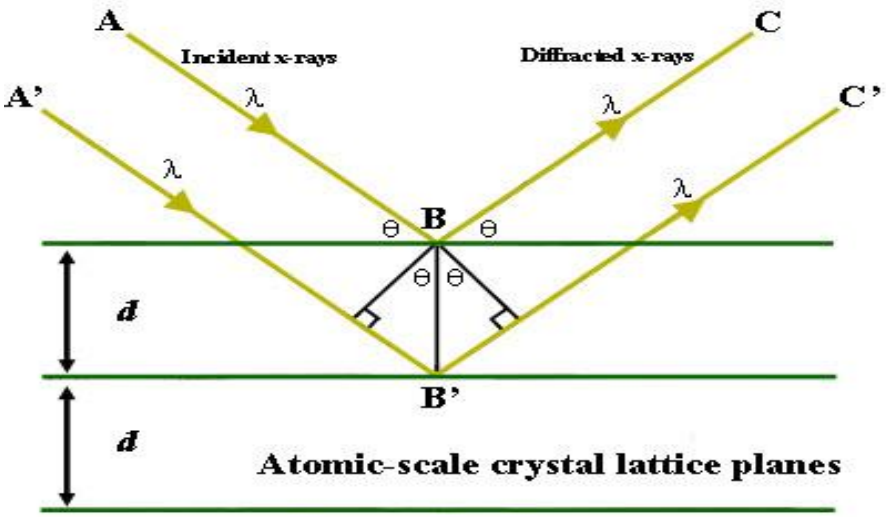


Fig.3

nλ= 2dsinθ---------- (3)

Here, λ=wavelength of the X-ray radiation and equal to 1.54 Å

d= distance of crystal layer

θ= incident angle

n= odder of diffraction

From eq. (3)

$$d=\frac{n\lambda}{2sin\theta}$$

Where n=1 and λ=1.54

$$d=\frac{1\times 1.54}{2\times sin21.63}$$

$$d=\frac{1.54}{2\times 0.36861}$$

$$d=\frac{1.54}{0.73722}$$

d = 2.088 A^0..... (4)

Put eq. (4) in eq. (2)

$$\frac{1}{(2.088)^2} = \frac{4}{a^2}$$

$a^2 = 4 \times 4.359$

$a^2 = 17.436$

$a = \sqrt{17.436}$

a = 4.175Å

Hence, the lattice parameter 'a' is = 4.175Å

CONCLUSIONS

Nanocrystalline NiO nanoparticles were synthesised by inexpensive sol gel method. The identification of cubic structure of NiO nanocrystallite is confirmed. X-ray diffraction (XRD) results indicates that the crystallite size is in 20-30nm range with average size of 25nm, lattice parameter a= 4.175A^0 for cubic structure of NiO. Here NiO nanoparticles were annealed at temperature 700˚C.

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