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Analysis of Structure and Surface Morphology, Synthesis of Nickel Oxide Nanoparticles by Hydrothermal Method

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ABSTRACT

Nickel Oxide (NiO) nanoparticles were synthesized by the hydrothermal method using Nickel Chloride Hexahydrate (NiCl₂.6H₂O) as the precursor. The synthesized precursor was calcined at 300°C for 4 hours. The XRD analysis depicts the calcined sample has a cubic structure with an average crystal size of around 18.02 nm. The FTIR analysis ensures the presence of a functional group of nickel oxide. The UV analysis presents a bandgap of 3.8 eV. And the SEM analysis confirms the morphology.

Keywords:- NiO nanoparticles, XRD, FTIR, UV, SEM

I. INTRODUCTION

Nano-particle oxides of transition metals have influenced researchers. These materials have unique properties which are uses in many advanced applications [1-3]. Nano-structured nickel oxide is a good example having a large exciton binding energy and a wide band gap ranging from 3.6 to 4.0eV[4,5]. This p-type semiconductor canbe used in many fields as optical, electronic, catalytic and super-paramagnetic devices like transparent conductor films, gas sensors, alkaline battery cathodes, dye-sensitized solar cells and solid oxide fuel cells[4-11].

II. EXPERIMENTAL

2.1 Chemical Used



FIGURE.1 Photograph of synthesis of NiO

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The Molychem Nickel Chloride Hexahidrate (NiCl₂.6H₂O) chemical, the Merck Polyethylene Glycol (PEG) solution, thePaskem Ammonia (NH₃)solution and the PBPL distilled water as dispersing solvent were used as the raw material in the synthesis of Nickel Oxide (NiO).

2.2 Synthesis of Nickel Oxide:-

Nickel oxide (NiO) nanoparticles were prepared by hydrothermal method under continuous stirring for 4 h at the room temperature. The Polyethylene Glycol and Ammoniawere used bydrop wise. The prepared solution was then kept into autoclave and heated at 100 °C for 15 h. After cooling, the light green precipitate was formed and washed with distilled water several times to remove impurities. The green precipitate was dried at Thermo Scientific HERATHERM Oven at 80°C for 10 hours. The obtained precursor was calcined at 300°C for 4 hours. The black nanoparticles were obtained and grinded as shown in FIGURE.1. The result was black Nanocrystalline powderreceived.

III. CHARACTERIZATION DETAILS

The calcined Nickel Oxide (NiO) nanoparticles were examined by Rigaku Smart Lab X-ray Diffractometer (XRD) with radiation source of wavelength $\lambda = 1.5406$ A°, scanned for a diffraction angle (20) in the range 20^o to 80^o to obtain crystal structure, phase composition, purity, crystalline size and average size. The BRUKER ALPHA II Fourier transform infrared spectrometer (FT-IR) confirms the existence of functional group of NiO and the spectrum of nickel oxide (as pellets in KBr) shows many bands ranges from (4000-400) cm⁻¹. The UV– Visible spectrum of NiO nanoparticles was measured by using aUV-PLUS UV VISIBLE SPECTROPHOTOMETER of MOTRAS SCIENTIFIC in the range 300-700nm and dispersion was in absolute ethanol to getabsorption edge and band gap. Scanning electron microscope (SEM) was used to identify the surface morphological behavior of Nickel oxide (NiO) nanoparticles.

IV. RESULTS AND DISCUSSION

4.1 X-ray diffraction (XRD) analysis:-

The Peak Position from 20 was obtained from FIGURE.2. The noticed diffraction peaks or Miller Indices are shown in Table 1. The standard structure is cubic with a=b=c=4.180 A° from JCPDS Card. The sharp and high-intensity peaks ensures that the synthesized NiO nanoparticles are crystalline in nature and there is no impurity peaks. To calculate d-spacing Bragg's equationis used:

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



FIGURE.2 XRD spectrum of NiO nanoparticles

Where d is d-spacing in A°, n is order of diffraction (1), λ is wavelength of x-ray (1.5406A°), θ is Bragg's diffraction angle. So, calculated value of d-spacing is correspondence to standard value from JCPDS Card. Most intense reflection {200} at 20=43.22° is selected. To calculate Lattice constant for cubic crystal system following formula is used:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

Where,d is d-spacing, h, k, l is miller indices and 'a' is lattice constant. So, calculated value of lattice constant is correspondence to the standard value from JCPDS Card. The Crystallite size was calculated by the Scherer equation:

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

Where,D is the crystallite size of the powder in nm, K is 0.9 (Scherer Constant), λ is 0.15406nm, the wavelength of the x-ray sources, β is the full width at half-maximum (FWHM) in radians, θ is Bragg's diffraction angle in radians. FWHM (β) was obtained from peak fitting. So, the value of β is 0.53503°, 0.46809°, 0.44051° and 0.58845°. So, the calculated value of D was 15.66nm, 18.25nm, 21.13nm and 17.05 nm. The average crystallite size isD=18.02nm.The average crystallite size of produced NiO powder confirms the formation of the nano-crystalline structure.

Planes	2θ values (Degree)		d-spacing values d(A°)		Lattice Constant a (A°)		FWHM
(hkl)	JCPDS	Experimental	JCPDS	Experimental	JCPDS	Experimental	В
111	37.22	37.18	2.41	2.48	4.180	4.259	0.53
200	43.25	43.22	2.09	2.13	4.180	4.259	0.46
220	62.82	62.82	1.47	1.48	4.180	4.259	0.44
311	75.34	75.36	1.26	1.26	4.180	4.259	0.58

Table 1 Compared values of miller indices, 20, d-spacing and lattice constant with JCPDS card.

4.2 Fourier transform infrared spectroscopy (FTIR) analysis:-



FIGURE.3 FTIR spectrum of NiO nanoparticles

FIGURE.3 shows the FTIR spectrum of NiO nanoparticles and presence of functional group of inorganic compounds. There are several absorption peaks exists in the spectrum of NiO nanoparticles at 3404cm⁻¹, 2925cm⁻¹, 1603cm⁻¹, 1389cm⁻¹, 1113cm⁻¹, 645cm⁻¹and (419 and 446) cm⁻¹. The peak at 3404 cm⁻¹ in the FT-IR spectrum is related to O-H bond. The bond at 2925 cm⁻¹ is correspondence to C-H stretching mode. The absorption at 1603 cm⁻¹ attributed to hydroxyl groups. The absorption bonds at 1389 cm⁻¹ and 1113 cm⁻¹ indicates the existence of carbonates. But absorption bond at 645 cm⁻¹ is assigned to Ni-O-H stretching bond. The absorption bonds at 419 and 446cm⁻¹ are associated to Ni-O vibration bond. The above information confirms the formation of pure NiO nanoparticles from reference [12] [13] [14].



4.3 UV-visible spectroscopy (UV-Vis) analysis



FIGURE.4 UV-visible spectrum of NiO nanoparticles and $(\alpha h\nu)^2$ vs. (hv) spectrum of NiO nanoparticles FIGURE.4 (a) shows the UV-visible spectrum of NiO nanoparticles after calcination in the range 300-700nm. The observed absorption edge is in the range 300-375nm. The blue shift of the absorptionedges is related to the size decrease of particles due to the quantum confinement effect of nanoparticlesfor different sized nanocrystals [14].The direct band gap values of the NiO samples were determined by Tauc's relation [15]:

 $\alpha h \upsilon = \alpha_{\circ} (h \upsilon - E_g)^{1/2}$

Where h ν , α_{\circ} and E_g are photon energy, a constant and optical band gap of the nanoparticles, respectively, α is absorption coefficient. Absorption coefficient (α), h ν , α h ν and $(\alpha$ h $\nu)^2$ of NiO nanoparticlescan be calculated from the absorption spectra. Then values of E_g were determined by extrapolations of the linear regions of the plot of $(\alpha$ h $\nu)^2$ versus E_g = h ν as shown in FIGURE.4 (b). The obtained energy band gap was found to be 3.8 eV for pure NiO.

4.4 Scanning electron microscopy (SEM) analysis:-

The morphology of NiO nanoparticles is shown in FIGURE 5 a, b, c. Figure 7 shows FESEM images of NiO nanoparticles at different magnification. So morphology was confirmed by FESEM analysis. Particles are agglomerated in nature it is clear from the images. Cause of overlapping or aggregating of smaller particles larger particles is also present. The images clearly show that randomly distributed grains with smaller size and the porous cubic topography particles as compared to XRD are observed. 1 μ m nano particle has 15.00 KX magnifications, 200 nm nano particles have 50.00 KX magnification and 20 nm nano particles have 200.00 KX magnification.



FIGURE.5 SEM images of NiO nanoparticles

V. CONCLUSION

The nanoparticles of NiO have been successfully synthesized by hydrothermal method and calcined at 300°C for 4 hours at room temperature. The formation of NiO nanoparticles by the hydrothermal method is simple, less dangerous, and has a short synthesized time. XRD analysis confirms the cubic structure and 18.02nm average crystal size. FTIR analysis presents the functional group of NiO and confirms the formation of NiO nanoparticles. In UV analysis, absorption edge is observed in the range 300-375nm and obtained energy band gap at 3.8 eV. And the SEM analysis confirms the agglomerate NiO nanoparticles forming porous cubic topography particles.

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