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SYNTHESIS AND CHARACTERIZATION OF COBALT HYDROXIDE NANOPARTICLES

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Abstract:-Cobalt hydroxide nanoparticles were synthesized via chemical co-precipitation method from cobalt chloride and sodium hydroxide. Structural and compositional properties were characterized by XRD, SEM, FTIR and UV spectroscopy. X-ray diffraction (XRD) confirmed the preferential growth of cobalt hydroxide nanoparticles that width is 73.42nm. The SEM image shows the synthesized cobalt hydroxide show well crystallized particles with rod like morphology. The FTIR spectrum is used to study the stretching and bending frequencies of molecular functional groups in the sample. From UV spectrum, the band gap of cobalt hydroxide nanoparticles is found to be 2.7eV.

Keywords:XRD, SEM, FTIR, UV.

1.INTRODUCTION

Research on layered transition-metal hydroxide materials such as nickel hydroxide and cobalt hydroxide has received considerable attention in recent years due to these materials potential applications as catalysts, supports, anion adsorbants, magnetic materials and ion exchangers as well as high-performance electrode materials of alkaline secondary batteries and supercapacitors1-7. Among a variety of layered transition-metal hydroxide materials,cobalt hydroxides have attracted interest due to their layered structure with large interlayer spacing, their high electrochemical redox activity, and the possibility of enhanced performance depending upon preparation conditions Cobalt hydroxide is used as a drier for paints and varnishes and is added to lithographic printing inks to enhance their drying properties. Other applications are in the preparation of cobalt salts, as a catalyst, and in storage battery electrodes.

In this paper we have reported the synthesis of cobalt hydroxide nanoparticles through the chemical co-precipitation method.

2 .EXPERIMENTAL DETAILS

Nanoparticles of cobalt hydroxide were prepared by chemical co-precipitation method by adding cobalt chloride and sodium hydroxide. Precise amounts of reagents taking into account their purity were weighed and dissolved separately in distilled water into 0.1M concentration. After obtaining a homogeneous solution, the reagents were mixed using magnetic stirring. The precipitate was separated from the reaction mixture and washed several times with distilled water and ethanol. The wet precipitate was dried and thoroughly ground using agate mortar to obtain the samples in the form of fine powder.

3. TESTS CONDUCTED

X-ray diffraction is an ideal technique for the determination of crystallite size of the powder samples. The basic principle for such a determination involves precise quantification of the broadening of the peaks. XRD line broadening method of particle size estimation was chosen in this investigation for determining the crystallite size of the powder sample. XRD study of the powder sample was carried out at Alagappa University, Karaikudi. The

morphology of the powder sample was studied by the scanning electron microscope (SEM) analysis taken at STIC Cochin. The infra red spectroscopic (IR) studies of cobalt hydroxide nano particles were made by using 'SHIMADZU' FTIR 8400S model spectrometer through KBr method. The UV spectrum was taken in the absorbance mode in the wavelength range from 200 to 800 nm.

4. RESULTS AND DISCUSSION

4.1. XRD studies

4.1.1. XRD – Particle Size Calculation

The XRD patterns of the prepared samples of cobalt hydroxide nanoparticles are shown in figure.1. XRD studies reveal that the samples are nano sized and crystalline. The fine particle nature of the samples is reflected in the X-ray line broadening.

The size of the synthesized cobalt hydroxide nano particles are calculated using Scherrer equation.

$$D = 0.9?/\beta \cos\theta \qquad (1)?$$

where λ represents wavelength of X rays, ß represents half width at full maximum and ? is the diffraction angle. The average grain size of the particles is found to be 73.42nm. The peak list in the XRD pattern is given in table-1.

Table-1.Intensity of XRD peaks.

2θ of peak(deg)	Height(counts)	FWHM(deg)	d- spacing (Å)	Relative intensity(%)
18.97	58	0.11	4.64794	100
51.24	43	0.10	1.7786	75.03

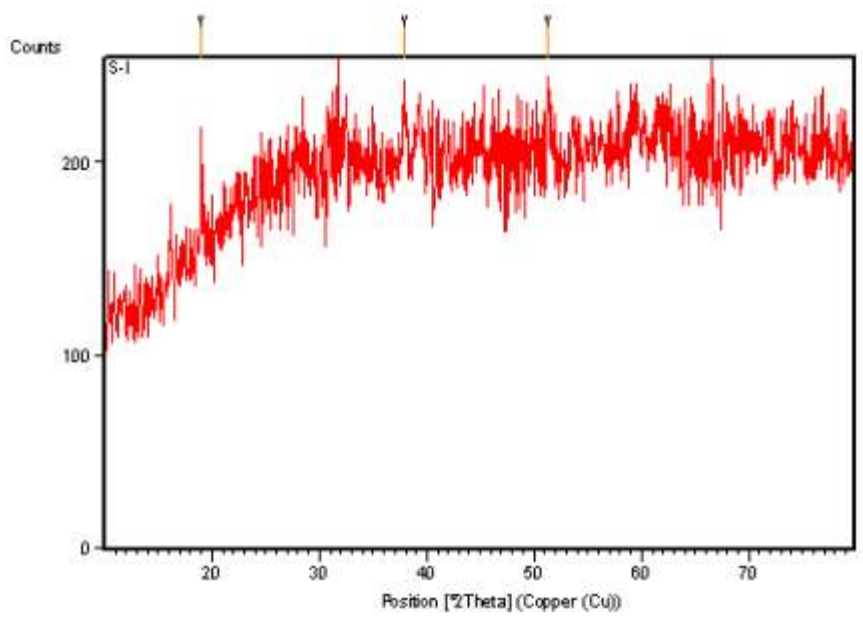

Figure.1 XRD pattern of cobalt hydroxide nano particles.

A good agreement between the Experimental diffraction angle [2θ] and Standard diffraction angle [2?] of specimen is confirming standard of the specimen. Two peaks at 2? values of cobalt hydroxide is observed and tabulated in table-2 and compared with the standard powder diffraction card of Joint Committee on Powder

Diffraction Standards (JCPDS), cobalt hydroxide file No. 47-1734. The d-spacing values of experimental is also confirming to the standard values.

Table.2. Experimental and standard diffraction angles of cobalt hydroxide specimen.

Experimental		Standard- JCPDS 47-1734	
Diffraction angle (2q in degrees)	D spacing (A°)	Diffraction angle (2q in degrees)	D spacing(A°)
18.97	4.64794	19.058	4.6530
51.24	1.7786	51.328	1.7786

4.1.2. XRD - Expected 2θ Positions

The value of d (the interplanar spacing between the atoms) is calculated using

Bragg's Law: 2dsinθ = n? (2)

$$d = \frac{\lambda}{2Sin\theta}(n = 1) \qquad (3)$$

Wavelength λ = 1.5418 Å for Cu Ka

The expected 2? positions of all the peaks in the diffraction pattern and the interplanar Spacing d for each peak is calculated using following formula and the details are shown in table-3.

$$\frac{1}{d2} = \frac{4}{3}\frac{h2 + hk + k2}{a2} + \frac{l2}{c2} \qquad (4)$$

Bragg's Law is used to determine the 2θ value: The expected 2? and d values are close with the experimental 2? and d values.

Table.3 The Lattice plane and the lattice spacing from d from XRD of cobalt hydroxide nanoparticles

hkl	2θ(deg)		D((Å)	
	Experiment	Expected	Experiment	Expected
001	18.97	19.058	4.64794	4.6530
012	51.24	51.328	1.7786	1.7786

4.1.3. XRD – Dislocation Density

The dislocation density is defined as the length of dislocation lines per unit volume of the crystal. In materials science, a dislocation is a crystallographic defect, or irregularity, within a crystal structure. The presence of dislocations strongly influences many of the properties of materials. The movement of a dislocation is impeded by other dislocations present in the sample. Thus, a larger dislocation density implies a larger hardness.

The X-ray line profile analysis has been used to determine the dislocation density. The dislocation density (δ) in the sample has been determined using expression.

The dislocation density can be calculated from

$$\delta = \frac{1}{D^2} \qquad (5)$$

Where δ is dislocation density and D is the crystallite size. Results of the dislocation density calculated are given in table-4. The number of unit cell is calculated from

$$n = \pi (4/3) \times (D/2)^3 \times (1/V) \qquad (6)$$

Where D is the crystallite size and V is the cell volume of the sample [8].

Table-4. Dislocation Density and Number of Unit Cell from XRD.

2θ (deg)	Particle Size D (nm)	Dislocation Density (m^2) $\delta = 1 / D^2$ $x10^{14}$	Number of Unit Cell $x10^5$
18.97	73.42	1.85512	50.640
51.24	80.30	1.5585	66.2524

It is observed from these tabulated details, dislocation density is indirectly proportional to particle size and number of unit cell. Dislocation density increases while both particle size and number of unit cell decreases.

4.1.4. XRD – Morphology Index

A XRD morphology index (MI) is calculated from FWHM of XRD data using the relation

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p} \qquad (7)$$

Where M.I. is morphology index, FWHMh is highest FWHM value obtained from peaks and FWHMp is value of particular peak's FWHM for which M.I. is to be calculated. The relation between morphology index and particle size is shown in table-5.

Table-5. Relation between Morphology Index and Particle size.

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.00191889	73.42	0.5
0.001744	80.30	0.5230

It is observed that MI increases with particle size.

4.1.5 XRD - Crystallinity Index

It is generally agreed that the peak breadth of a specific phase of material is directly proportional to the mean crystallite size of that material. Quantitatively speaking, sharper XRD peaks are typically indicative of high nano crystalline nature and larger crystallite materials. From our XRD data, a peak broadening of the nanoparticles is noticed. The average particle size, as determined using the Scherrer equation, is calculated to be 73.42nm. Crystallinity index equation is given by

Icry = Dp (SEM, TEM) / Dcry (XRD) (Icry >= 1.00)

Where Icry is the crystallinity index; Dp is the particle size (obtained from either TEM or SEM (morphological analysis); Dcry is the particle size (calculated from the Scherrer equation). If Icry value is close to 1, then it is assumed that the crystallite size represents monocrystalline whereas a polycrystalline have a much larger crystallinity index [8]. The crystallinity index of the sample is 2.93 which is more than 1.0. The details are enumerated in Table-6.

Table-6. The crystallinity index of cobalt hydroxide nanoparticles

Sample	Dp (nm)	Dcry (nm)	Icry (unitless)	Particle Type
Cobalt hydroxide Nanoparticles	215.41	73.42	2.93	Polycrystalline

4.1.5. XRD – Unit Cell Parameters

Unit cell parameters values calculated from XRD are enumerated in table-7

Table-7. XRD parameters of cobalt hydroxide nanoparticles.

Parameters	Values
Structure	Primitive
Space group	$P_3ml(164)$
Symmetry of lattice	Hexagonal
Particle size	73.42 nm
Lattice parameters	a = 3.186; c = 4.653
Vol.unit cell(V)	40.90
Density (ρ)	3.773
Dislocation Density	1.85512×10^{14}
Mass	92.95amu

4.2. SEM studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized cobalt hydroxide nanoparticles. figure.2, figure.3 figure.4 and figure.5 show the SEM images of the cobalt hydroxide nanoparticles at various magnifications. The SEM images of cobalt hydroxide nanoparticles show well crystallized particles with rod shaped morphology. In this case the particles sizes are slightly increased and is also observed that the particles are distributed with agglomeration.

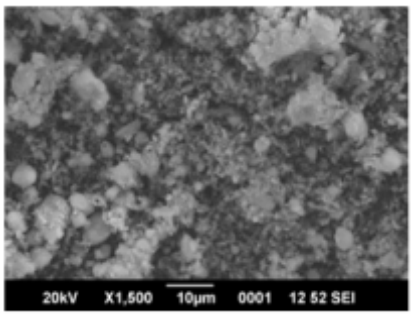

Figure.2 SEM image at 1500 magnifications.

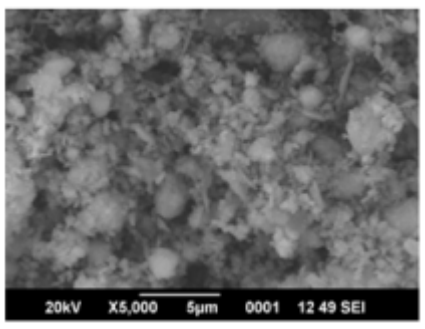

Figure.3 SEM image at 5000 magnifications.

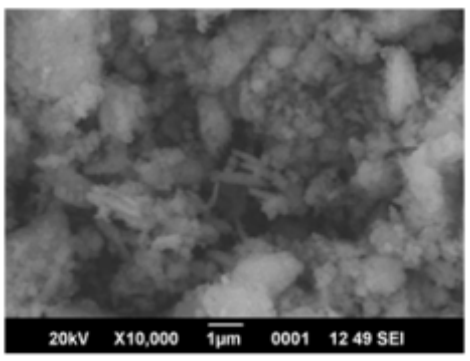

Figure.4 SEM image at 10000 magnifications.

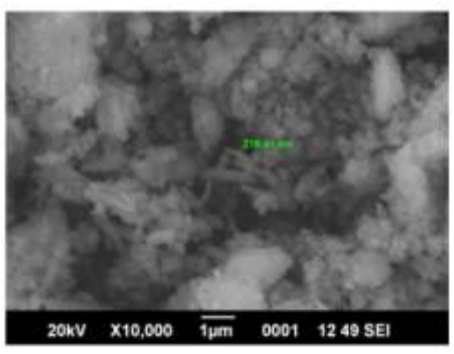

Figure.5 SEM image at 10000 magnifications.

4.3. FTIR Studies

The FTIR spectrum of the cobalt hydroxide sample is shown in the figure.9.The FTIR spectrum for cobalt hydroxide shows a strong peak at 3626.17 cm^{-1} corresponding to the free O-H group[9] and the peak at 1699.29 cm^{-1} , 1681.93 cm^{-1} , 1649.14 cm^{-1} , 1622.13 cm^{-1} , 1543.05 cm^{-1} and 1516.05 cm^{-1} are due to the bending mode of the hydroxyl group of water [10]. The spectrum also shows peak at 731.02 cm^{-1} , 499.56 cm^{-1} , 424.34 cm^{-1} which are below 800 $cm^{?1}$**,** indicating the absorptions are associated with Co-O stretching and Co-OH bending vibrations[11].

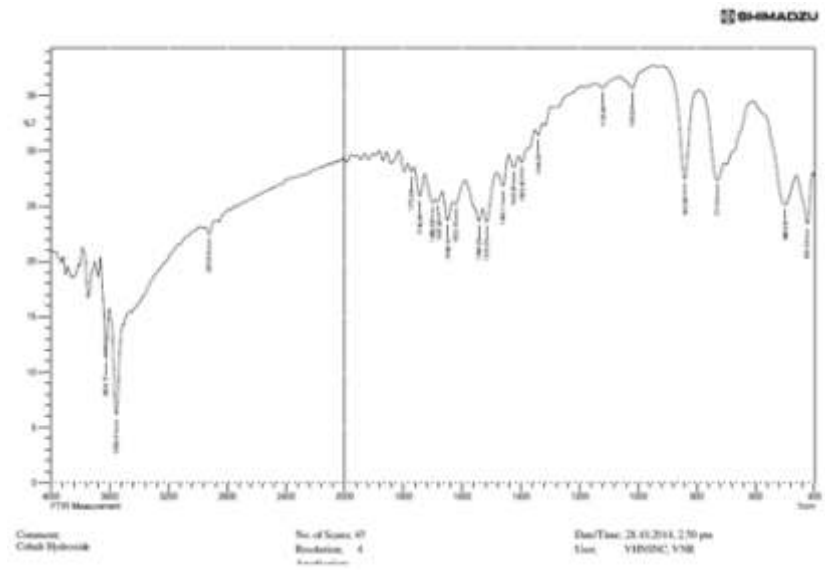

Figure.9 FTIR spectra of cobalt hydroxide nanoparticles.

4.4. UV Studies

The band gap of the prepared sample cobalt hydroxide was determined by using UV visible studies. From the UV spectrum the optical band gap of cobalt hydroxide is 2.7eV. Figure.10 shows the graph to find the band gap of cobalt hydroxide.

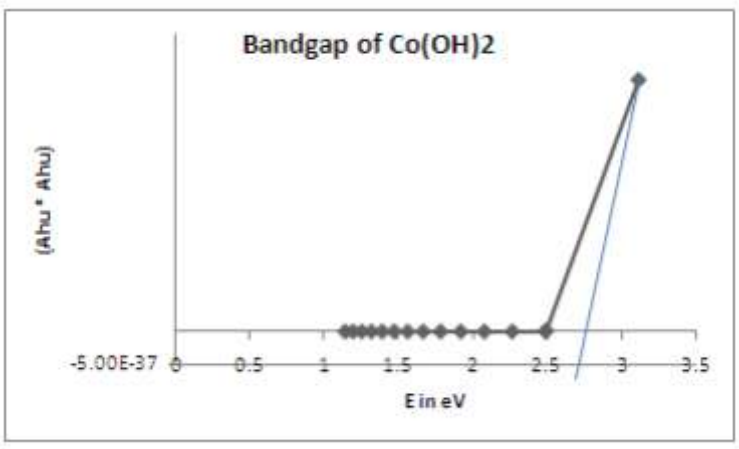

Figure.10 Graph to find the band gap of cobalt hydroxide nanoparticles.

5. CONCLUSIONS

The cobalt hydroxide nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (73.42nm). The SEM picture reveals the well crystallized particles with rod-like morphology. From the FTIR spectrum, the stretching and bending frequencies of the molecular functional groups in the sample are studied. From the UV spectra, the band gap was found.

6. REFERENCES

1.Nasirian A, Synthesis and characterization of Cu nanoparticles and studying of their catalytic properties. Int.J.Nano Dim. 2012; 2: 159-164.
2.Liu X, Geng B, Du Q, Ma J and Liu X, Temperature-controlled self-assembled synthesis of CuO Cu2O and Cu nanoparticles through a single-precursor route. Material science and engineering A. 2007; 448: 7-14.
3.Sun S , Murry C B , Weller D, Folks L and Moster A, On the synergistic coupling properties of composite CdS / TiO2 nano particle arrays confined in nano patterned hybrid thin films . Science. 2000; 287: 1989.
4.Wang J F, Gudiksen M S, Duan X F and Cui Lieber Y C M , Highly Polarized Photoluminescence and Photo detection from Single Indium Phosphide Nano wires. Science. 2001; 293: 1455.
5.Yu W D and Vivian W Y, Controlled Synthesis of Mono disperse Silver Nano cubes in Water. J. Am. Chem. Soc. 2004; 126: 13200.
6.Petit C, Taleb A and Pileni M P, Self -Organization of Magnetic Nanosized Cobalt Particles. Adv. Mater. 1998; 10: 259.
7.Taleb A, Petit C and Pileni M P, Optical Properties of self assembled 2D and 3D Superlattices of Silver Nanoparticles. Phys. Chem. 1998; 102: 2214.
8.Theivasanthi T and Alagar M, Konjac Biomolecules Assisted–Rod/ Spherical Shaped Lead Nano Powder Synthesized by Electrolytic Process and Its Characterization Studies. Nano Biomed. Eng. 2013; 5(1): 11-19.
9.Jhon Mauricio Aguirre A D, Adamo Gutiérrezb D and Oscar iraldo C D , Simple Route for the Synthesis of Copper Hydroxy Salts. J. Braz. Chem. Soc. 2011; 22: 546-551.
10.Frost R L and Kloprogge J T, Infrared emission spectroscopic study of brucite. Spectrochim. Acta Part A. 1999; 55: 2195-2205.
11.Sang Wook Kim, Bob Jin Kwon, Jeong Hoon Park, Min Goo Hur, Seung Dae yang and Hyun Jung, γ-ray Radiation Induced Synthesis and Characterization of a-Cobalt Hydroxide Nanoparticles, 910 Bull. Korean Chem. Soc. 2010, 31(4): 910-914.

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