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Synthesis and Characterisation of Iron II Hydroxide Nano Particles

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ABSTRACT

Iron (II) Hydroxide nano particles were synthesized via chemical co precipitation method from Iron (II) 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 Iron (II) Hydroxide nano particles that width is 30.91nm. The SEM image shows the synthesized Iron (II) Hydroxide show well crystallized particles with spherical 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 Iron (II) hydroxide nano particles is found to be 3.5eV. From AAS studies it is found that the absorbance is directly proportional to the concentration. The linear fit indicates that Iron (II) Hydroxide nanoparticles have been distributed in proper proportion.

1.Introduction

Nanotechnology is the engineering and art of manipulating matter at the nanoscale (1–100 nm) [1–3]. For environmental applications, nanotechnology offers the potential of novel functional materials, processes and devices with unique activity toward recalcitrant contaminants, enhanced mobility in environmental media and desired application flexibility [3–10].Many nano-based environmental technologies (e.g., sensors, sorbents, reactants) are under very active research and development, and are expected to emerge as the next generation environmental technologies to improve or replace various conventional environmental technologies in the near future[3–10].

In this paper we have reported the synthesis of Iron (II) Hydroxide nano particles through the chemical co-precipitation method. Anions such as selenite and selenate can be easily adsorbed on the positively charged surface of iron (II) hydroxide where they are subsequently reduced by Fe^{2+}. The resulting products are poorly soluble. Iron (II) hydroxide has also been investigated as an agent for the removal of toxic selenate and selenite ions from water systems such as wetlands. The iron (II) hydroxide reduces these ions to elemental selenium, which is insoluble in water and precipitates out. This means it has a low tendency to dissolve, but is not entirely insoluble. An acidic solution would allow this to disassociate more because the H^+ would react with the OH^- in the compound. In a basic solution iron (II) hydroxide is the electrochemically active material of the negative electrode of the nickel-iron battery.

2. Experimental Details

Nano particles of Iron (II) Hydroxide were prepared by chemical co precipitation method by adding Iron (II) 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 samples was carried out at Centre for Electro Chemical Research Institute, Karaikudi. The morphology of the powder samples was studied by the scanning electron microscope (SEM) analysis taken at STIC Cochin. The infra red spectroscopic (IR) studies of Copper 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 Iron (II) 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 Iron (II) hydroxide nano particles are calculated using Scherrer equation

$$D = 0.9\lambda/\beta \cos\theta$$

where λ represents wavelength of X rays, β represents half width at full maximum and θ is the diffraction angle.

2Theta = 32.2432; β = 0.2676 x 3.14 / 180 = 0.0046681; D = (0.9 * 0.154) / (0.0046681) * cos (16.1216) = 30.91nm

The average grain size of the particles is found to be 30.91nm. The peak list in the XRD pattern is given in table-1.

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Table-1.Intensity of XRD peaks

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
32.2432	553.11	0.2676	2.77639	100.00
37.2195	25.66	0.2676	2.41582	4.64
66.6987	29.36	0.5353	1.40236	5.31

A good agreement between the Experimental diffraction angle [2θ] and Standard diffraction angle [2θ] of specimen is confirming standard of the specimen. Two peaks at 2θ values of Iron (II) Hydroxide is observed and tabulated in table-2 and compared with the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), Iron (II) Hydroxide file No. 13-0089. The d-spacing values of experimental is also confirming to the standard values.

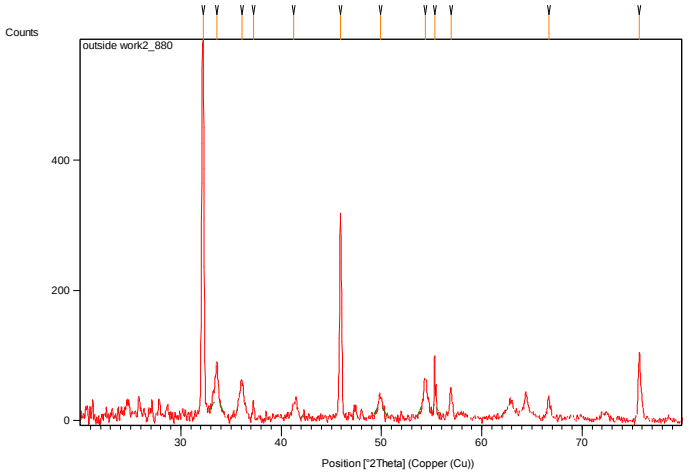

Figure.1 XRD pattern of Iron (II) Hydroxide Nano particles.

Table.2. Experimental and standard diffraction angles of Iron (II) Hydroxide specimen

Experimental		Standard – JCPDS 13-0089	
Diffraction angle (2θ in degrees)	D spacing (Å)	Diffraction angle (2θ in degrees)	D spacing (Å)
37.2195	2.41582	37.054	2.8170
66.6987	1.40236	66.662	1.6290

4.1.2. 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.

$$\delta = \frac{15\,\beta\,COS\theta}{4aD}$$

Where δ is dislocation density, β is broadening of diffraction line measured at half of its maximum intensity (in radian), θ is Bragg's diffraction angle (in degree), a is lattice constant (in nm) and D is particle size (in nm). The dislocation density can also be calculated from

$$\delta = \frac{1}{D^2}$$

Where δ is dislocation density and D is the crystallite size. Results of the dislocation density calculated from both the formulas are given in table-3. The number of unit cell is calculated from

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

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

Table-3. Dislocation Density and Number of Unit Cell from XRD.

2θ (deg)	Particle Size D (nm)	Dislocation Density (m^2)		Number of Unit Cell
		$\delta = 15\beta\cos\theta /4aD$	$\delta = 1 / D^2$	
32.2342	16.1216	1.7459×10^{15}	1.0469×10^{15}	3.6499×10^{5}
37.2195	31.43	1.62×10^{15}	1.01×10^{15}	3.8404×10^{5}
66.6987	17.83	5.02×10^{15}	3.14×10^{15}	0.7011×10^{5}

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 [12].

4.1.3. XRD – Morphology Index

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

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p}$$

Where M.I. is morphology index, $FWHM_h$ is highest FWHM value obtained from peaks and $FWHM_p$ 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-4.

Table-4. Relation between Morphology Index and Particle size

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.004688	30.9062	0.5
0.004413	31.4276	0.5151
0.007780	17.8264	0.3760

4.1.4 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 30.91nm. 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 [13]. The crystallinity index of the sample is 2.37 which is more than 1.0. The details are enumerated in Table-5.

Table-5. The crystallinity index of Iron (II) Hydroxide Nanoparticles

Sample	Dp (nm)	Dcry (nm)	Icry (unitless)	Particle Type
Iron (II) Hydroxide Nanoparticles	73.33	30.91	2.37	Polycrystalline

4.1.2. XRD – Unit Cell Parameters

Unit cell parameters values calculated from XRD are enumerated in table-6

Table-6. XRD parameters of Iron (II) Hydroxide Nanoparticles

Parameters	Values
Structure	Primitive
Space group	P3m1 (space group number : 164)
Symmetry of lattice	Orthorhombic
Particle size	30.91 nm
Lattice parameters	a = 3.258;c = 4.605
Vol.unit cell(V)	42.33
Mass	89.86amu

4.2. SEM studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized Iron (II) Hydroxide nano particles. figure.2, figure.3, figure.4 and figure.5 show the SEM images of the Iron (II) Hydroxide nano-particles at various magnifications. The SEM images of Iron (II) Hydroxide nano particles show well crystallized particles with spherical morphology. In this case the particles sizes are slightly increased and is also observed that the particles are distributed with agglomeration.

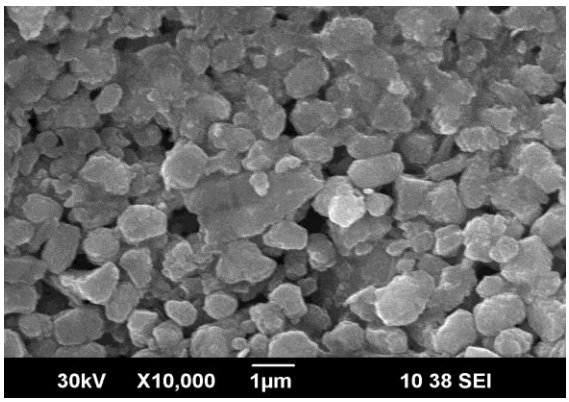

Figure.2 SEM image at 10000 magnifications.

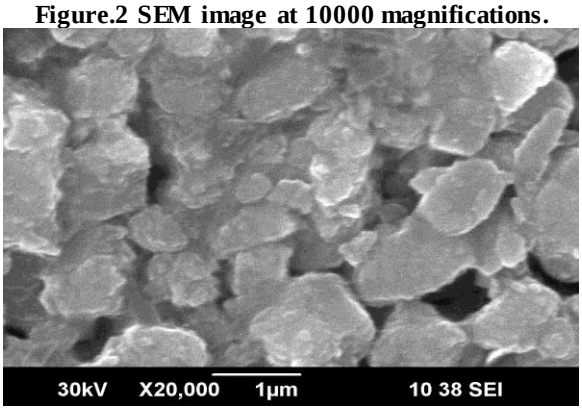

Figure.3 SEM image at 20000 magnifications.

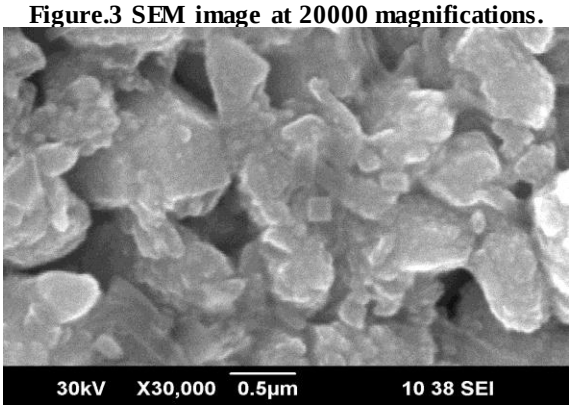

Figure.4 SEM image at 30000 magnifications.

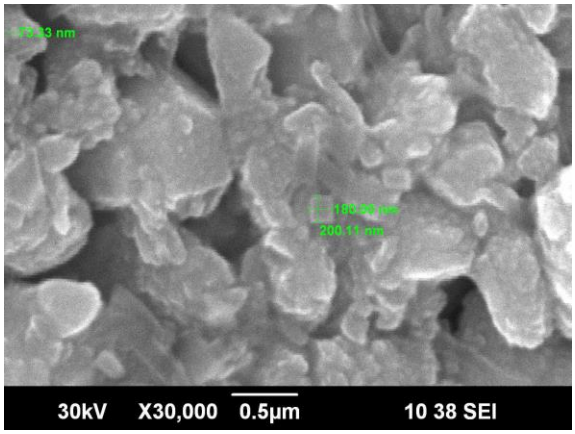

Figure.5 SEM image at 30000 magnifications.

4.3. FTIR Studies

The FTIR spectrum of the Iron (II) Hydroxide sample is shown in the figure.6.The FTIR spectrum for Iron (II) Hydroxide shows a strong peak at 3392.55cm^{-1} corresponding to the free O-H group .Another peak with a maximum of 1622.02cm-1 is due to the bending mode of the hydroxyl group of water. The spectrum also shows peak at 1400.22cm-1 indicating CH_2 bending and the peak at 478.31cm-1 indicates Fe-O vibrations [15].

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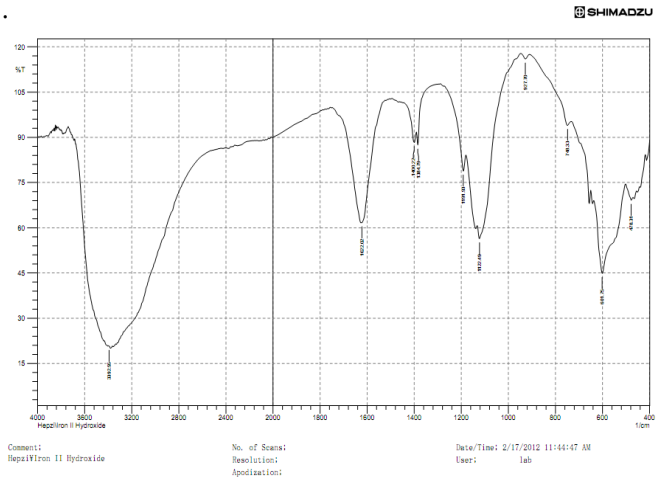

Figure.9 FTIR spectra of Iron (II) Hydroxide Nano particles

4.4. UV Studies

The band gap of the prepared sample Iron (II) Hydroxide was determined by using UV visible studies. From the UV spectrum the optical band gap of Iron (II) Hydroxide is 3.5eV. Figure.10 shows the graph to find the band gap of Iron (II) Hydroxide.

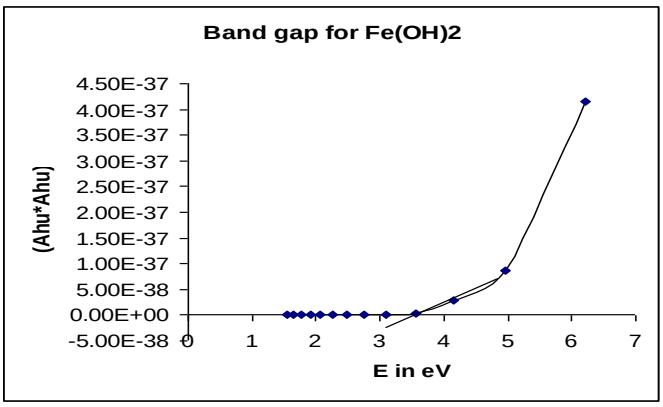

Figure.10 Graph to find the band gap of Iron (II) Hydroxide Nano particles

4.5. AAS Studies

The synthesized Iron (II) Hydroxide nanoparticles have been analyzed by AAS with optical parameter settings Fe wavelength 248.3nm and Air – C_2H_2 flame type. The results are given in table-7. A calibration curve diagram for Concentration of Iron (II) Hydroxide nanoparticles in parts per million (ppm) Vs Absorbance has been drawn and a linear fit has been got. It is observed from the figure.11 that the absorbance is directly proportional to the concentration. The linear fit indicates that Iron (II) Hydroxide nanoparticles have been distributed in proper proportion [14].

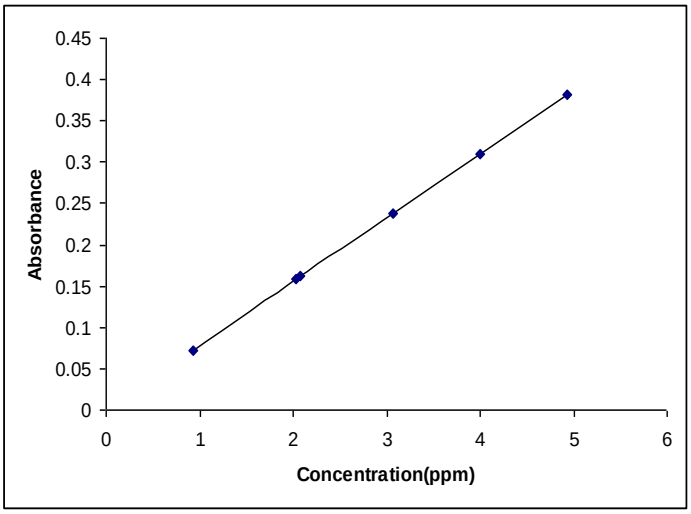

Figure. 11 Concentration Vs Absorbance.

Table-8 AAS Analysis Result.

True value	Standard	Sample	%R	Actual Concentration %	Concentration (ppm)	Absorbance
1.0000	0.9368	0.0726	94	40.1076	2.0375	0.1579
2.0000	2.0839	0.1615	104			
3.0000	3.0749	0.2383	102.3	Weight Factor = 1.000000 Volume Factor = 1.00 Dilution Factor = 1.00 Correction Factor = 1.000000		
4.0000	4.0014	0.3101	100			
5.0000	4.9330	0.3823	98.6			

5. Conclusions

The Iron (II) Hydroxide nano particles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (30.91nm). The SEM picture reveals the well crystallized particles with spherical 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. From AAS studies it is found that the absorbance is directly proportional to the concentration. The linear fit indicates that Iron (II) Hydroxide nanoparticles have been distributed in proper proportion.

6. References

1. Yuan-Pang Sun, Xiao-qin Li, Jiasheng Cao, Wei-xian Zhang, Paul Wang H, Characterization of zero-valent iron nanoparticles. Advances in Colloid and Interface Science. 2006; 120:47–56.

2. Brumfiel G. Nanotechnology: A little knowledge… Nature 2003; 424:246.

3. Mohamed H, Hassan A. Nanotechnology. Small things and big changes in the developing world Science. 2005; 309:65.

4. Zhang W. Nanoscale iron particles for environmental remediation: An overview J Nanopart Res 2003; 5:323- 332.

5. Wang C, Zhang W. Synthesizing nanoscale iron particles for rapid and complete dechlorination TCE and PCBs. Environ Sci Technol 1997; 31:2154.

6. Mahfuz U, Ahmed M. A review of nanoscale wireless sensor networks for environmental protection: Prospects and challenges. Sci Technol Adv Mater 2005; 6:302 - 306.

7. Rose-Pehrsson S, Pehrsson P. Nanotechnology and the environment. Washington, DC: American Chemical Society; 2005.

8. Abrams BL, Wilcoxon JP. Nanosize semiconductors for photo oxidation. Crit Rev Solid State Mater Sci. 2005; 30:153-182.

9. Yuan G. Part A: Toxic/Hazardous Substances & Environmental Engineering J Environ Sci Health 2004; 39:2661-2670.

10. Nagaveni K, Sivalingam G, Hegde S, Madras G. Photo catalytic degradation of organic compounds over combustion-synthesized nano-TiO2.Environ Sci Technol 2004; 38(5):1600-1604.

11. Modi A, Koratkar N, Lass E, Wei B, Ajayan P. Miniaturized gas ionization sensors using carbon nanotubes. Nature 2003; 424:171-174.

12. Alagar M, Theivasanthi T, Nano sized copper particles by electrolytic synthesis and characterizations. Int J Phy Sci. 2011; 6(15): 3662-3671.

13. Theivasanthi, 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.

14. Maria C, Antoni D, Wladimirsky A, Palacios D, Liliana C, Ana C, Gonzalez B, Enrique JB, Mercader RC, Spectroscopic investigations of iron(II) and iron(III) oxalates. J Braz Chem Soc, 2009; 20(3).