SYNTHESIS AND CHARACTERIZATION OF COBALT CARBONATE NANOPARTICLES

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ABSTRACT

Cobalt carbonate nanoparticles were synthesized via chemical co-precipitation method from cobalt chloride and sodium carbonate. The formed nanoparticle is characterized by powder x-ray diffraction, scanning electron microscopy, ultra-violet spectroscopy and fourier transform infrared spectroscopy, confirmed the preferential growth of cobalt carbonate nanoparticles that width is 63.69nm. The SEM image shows the synthesized cobalt carbonate 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 cobalt carbonate nanoparticles is found to be 5eV.

Keywords: FTIR, SEM, XRD, UV.

1. Introduction

Synthesizing metallic nanoparticles following wet-chemistry routes is a powerful way of obtaining a reproducible macroscopic amount of homogeneous sample. Several wet-chemical methods have been developed to synthesize cobalt crystals with different morphologies, for example, pyrolysis, solvothermal and hydrothermal decomposition, microfluidic synthesis, modified polyol processes, and template-based methods. It has been reported that liquid-phase reduction methods are relatively simple and do not require special equipment. Moreover, they are considered to be less expensive and quicker to implement, which are desirable qualities for future attempts of large-scale production. Much attention has been paid to the characteristics of cobalt nanoparticles; however, there has been little research

on the growth mechanism of cobalt nanoparticles. The shape and size of the nanoparticles influence the physical characterization of these novel materials [1].

Cobalt carbonate is a precursor to cobalt carbonyl and various cobalt salts. It is a component of dietary supplements since cobalt is an essential element. It is a precursor to blue pottery glazes, famously in the case of Delftware.

2. Experimental Details

Nanoparticles of cobalt carbonate were prepared by chemical co-precipitation method by adding cobalt chloride and sodium carbonate. 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 Alagappa University, 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 cobalt carbonate 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 - Crystalline Size

The XRD patterns of the prepared samples of cobalt carbonate 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 carbonate nanoparticles are calculated using Scherrer equation

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

where λ represents wavelength of X rays, β represents half width at full maximum and θ is the diffraction angle. The average grain size of the particles is found to be 63.69nm.

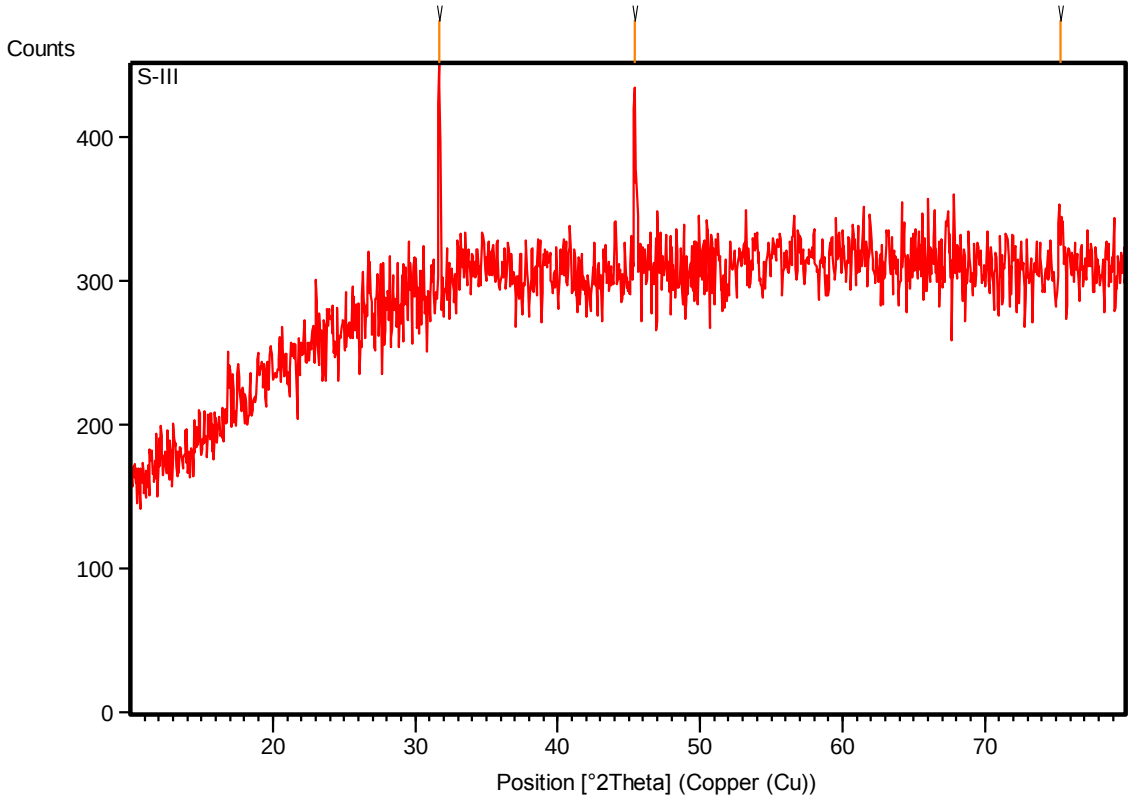

Figure.1 XRD Pattern of Cobalt Carbonate Nanoparticles

A good agreement between the experimental diffraction angle [2θ] and standard diffraction angle [2θ] of specimen is confirming standard of the specimen. The peaks at 2θ values of cobalt carbonate is observed and tabulated in table-1 and compared with the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), cobalt carbonate file No 11-0692. The d-spacing values of experimental is also confirming to the standard values.

Table.1. Experimental and Standard Diffraction Angles of Cobalt Carbonate Specimen.

Experimental		Standard – JCPDS 70-2053	
Diffraction angle (2θ in degrees)	D spacing (Å)	Diffraction angle (2θ in degrees)	D spacing (Å)
31.67	2.82286	32.617	2.7430
45.408	1.99576	46.583	1.9480
75.3	1.26138	76.309	1.2468

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 dislocation density can be calculated from

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

Where δ is dislocation density and D is the crystallite size. Results of the dislocation density calculated are given in table-2. The number of unit cell is calculated from

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

Where D is the crystallite size and V is the cell volume of the sample [2].

Table-2. Dislocation Density and Number of Unit Cell from XRD.

2θ (deg)	Particle Size D (nm)	Dislocation Density (m^2) $\delta = 1 / D^2 \times 10^{14}$	Number of Unit Cell $X10^5$
31.67	63.69	2.47	4.808
45.408	95.94	1.09	16.43
75.3	33.53	8.89	0.701

It is observed from these tabulated details, and from figures.2, 3 & 4, 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.

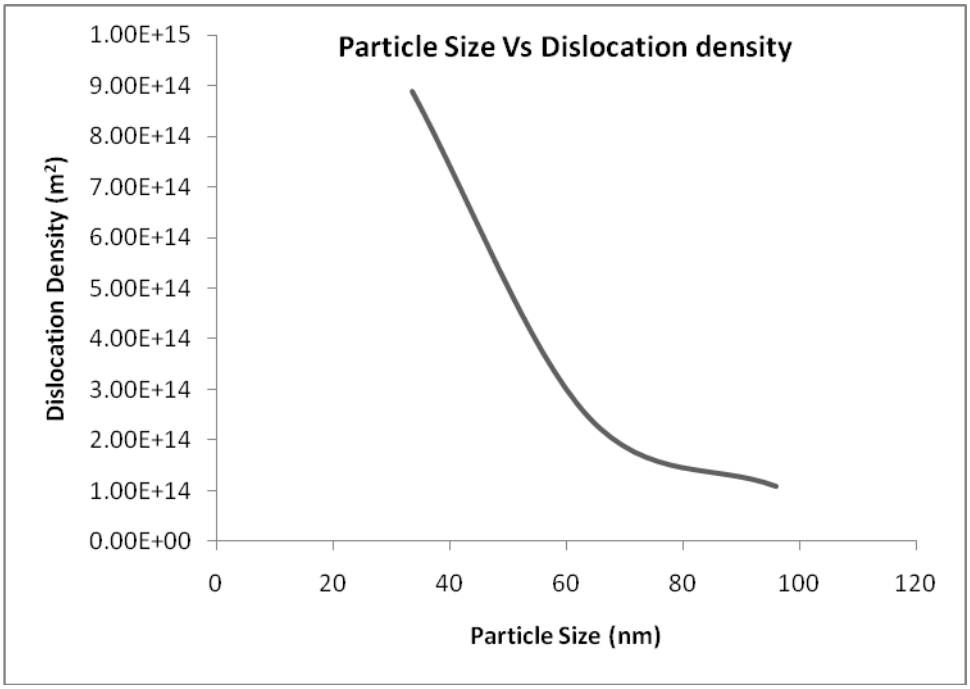

Figure.2 Particle size Vs Dislocation Density for Cobalt Carbonate Nanoparticles.

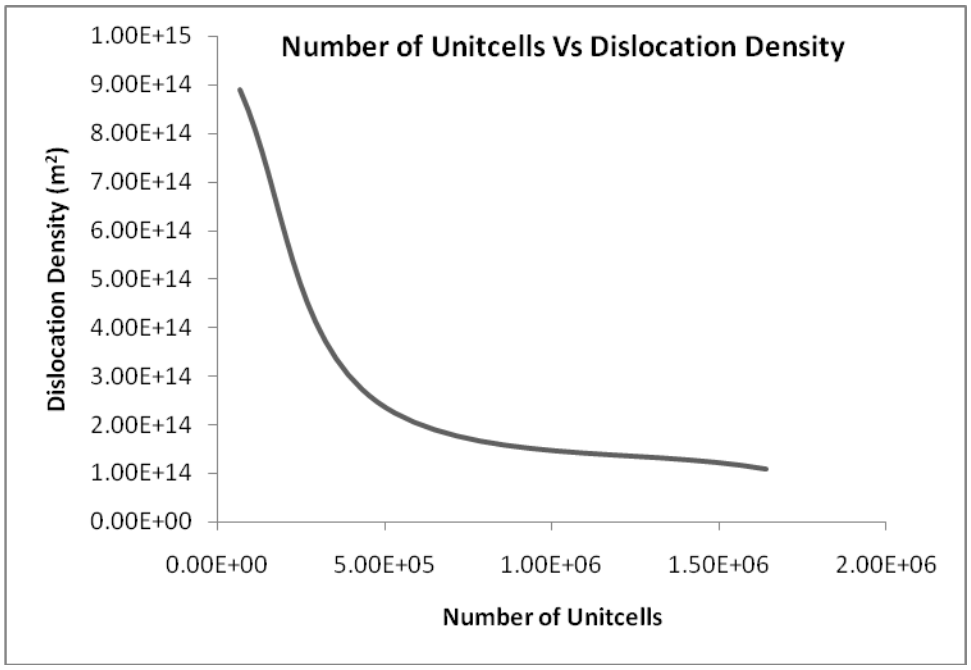

Figure.3 Number of Unit cells Vs Dislocation density for Cobalt Carbonate Nanoparticles.

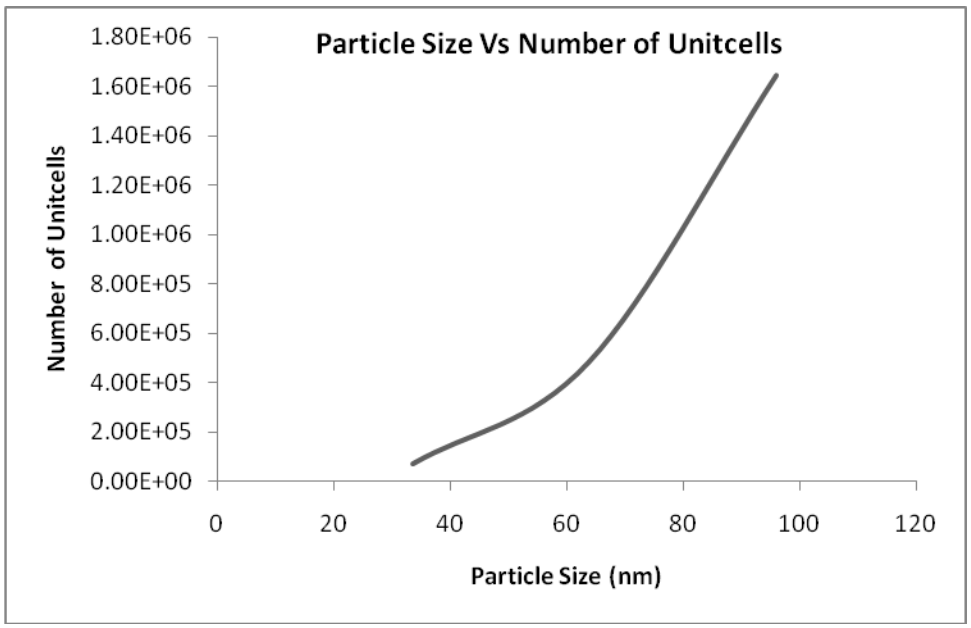

Figure.4 Particle size Vs Number of unit cells for Cobalt Carbonate Nanoparticles.

4.1.3. XRD – Morphology Index

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

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p} \quad (5)$$

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-3.

Table 3. Relation between Morphology Index and Particle size for Cobalt Carbonate Nanoparticles.

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.0027	63.69	0.5
0.00157	95.94	0.6323
0.0052	33.53	0.3418

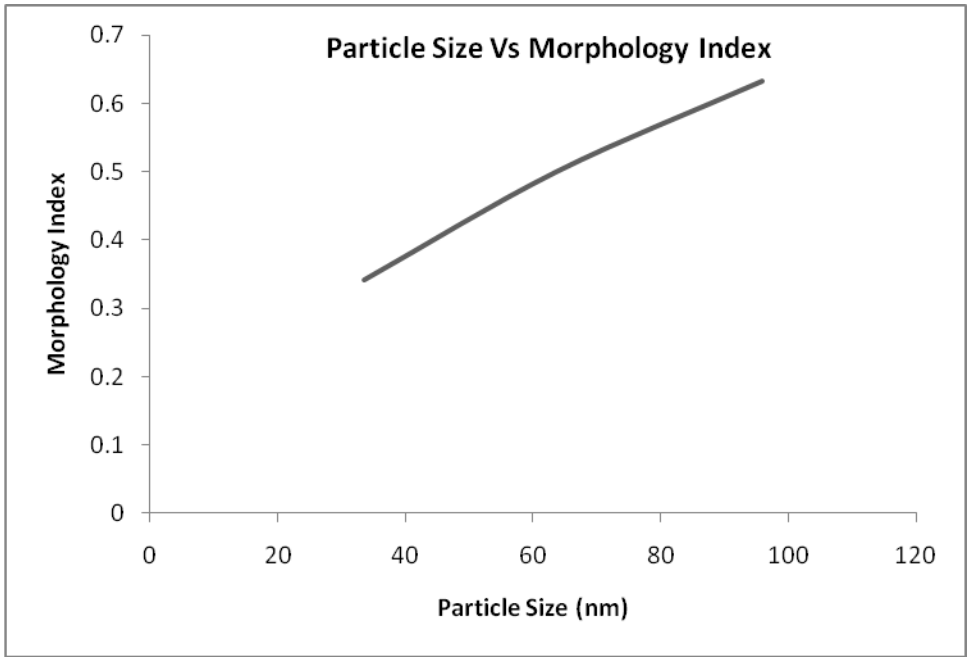

Figure.5 Morphology Index of Cobalt Carbonate Nanoparticles.

It is observed that MI has direct relationship with particle size and the results are shown in Figure.5.

4.1.4. XRD – Unit Cell Parameters

Unit cell parameters values calculated from XRD are enumerated in table-4.

Table-4. XRD parameters of Cobalt Carbonate nanoparticles.

Parameters	Values
Structure	Rhomb- centered
Space group	R3C(167)
Symmetry of lattice	Rhombohedral
Particle size	63.69 nm
Lattice parameters	a = 4.659;c = 14.957
Vol.unit cell(V)	281.16
Density (ρ)	4.215
Dislocation Density	$2.47x10^{14}$
Mass	118.94amu

4.2. SEM studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized cobalt carbonate nanoparticles. Figure.6, Figure.7 and Figure.8 show the SEM images of the cobalt carbonate nanoparticles at various magnifications. The SEM images of cobalt carbonate nanoparticles 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.

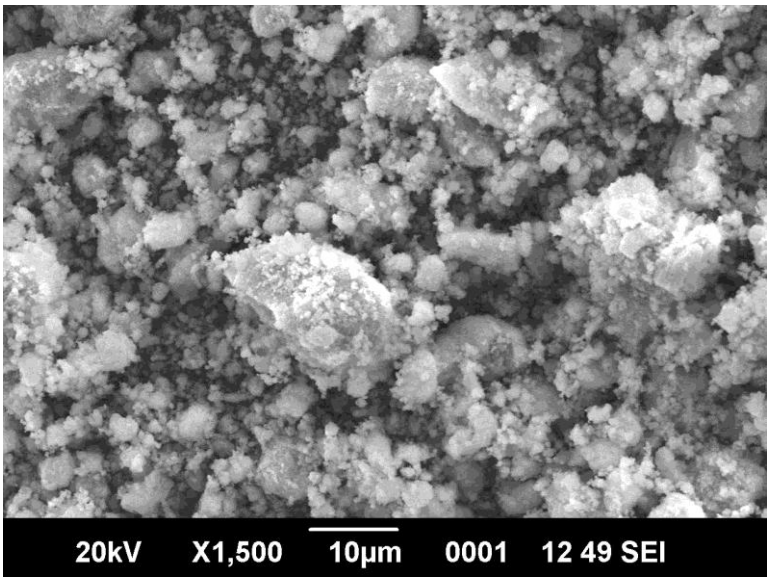

Figure.6 SEM image at 1500 Magnifications

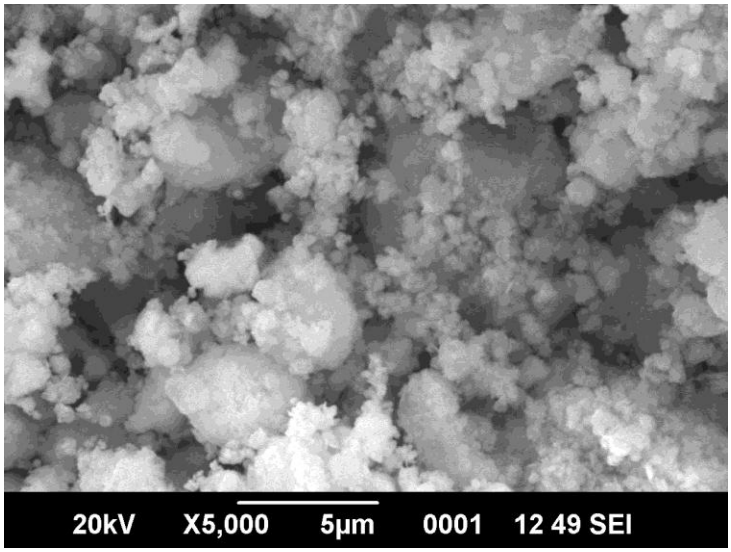

Figure.7 SEM image at 5000 Magnifications

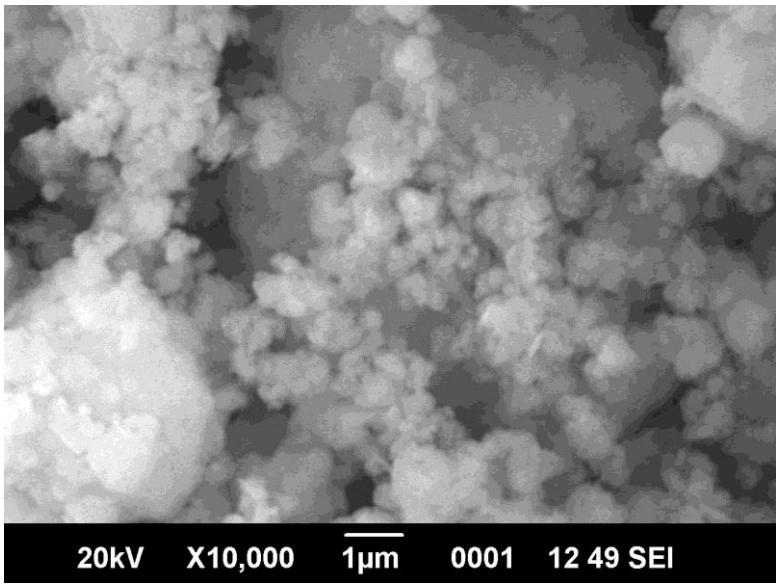

Figure.8 SEM image at 10000 Magnifications

4.3. FTIR Studies

The FTIR spectrum of the cobalt carbonate sample is shown in the figure.9.The FTIR spectrum for cobalt carbonate show peaks at 3500.80 cm^{-1} corresponding to the free O-H group [3] and the peak at 1647.21 cm^{-1} is bending mode of hydroxyl group [3]. The peak at 833.25cm^{-1} is due to carbonate ions [4] and the peak at 420.48 cm^{-1} and 513.07 cm^{-1} corresponds to the Co-O bond and 673.16 cm^{-1} was assigned to the bridging vibration of O-Co-O bond [5].

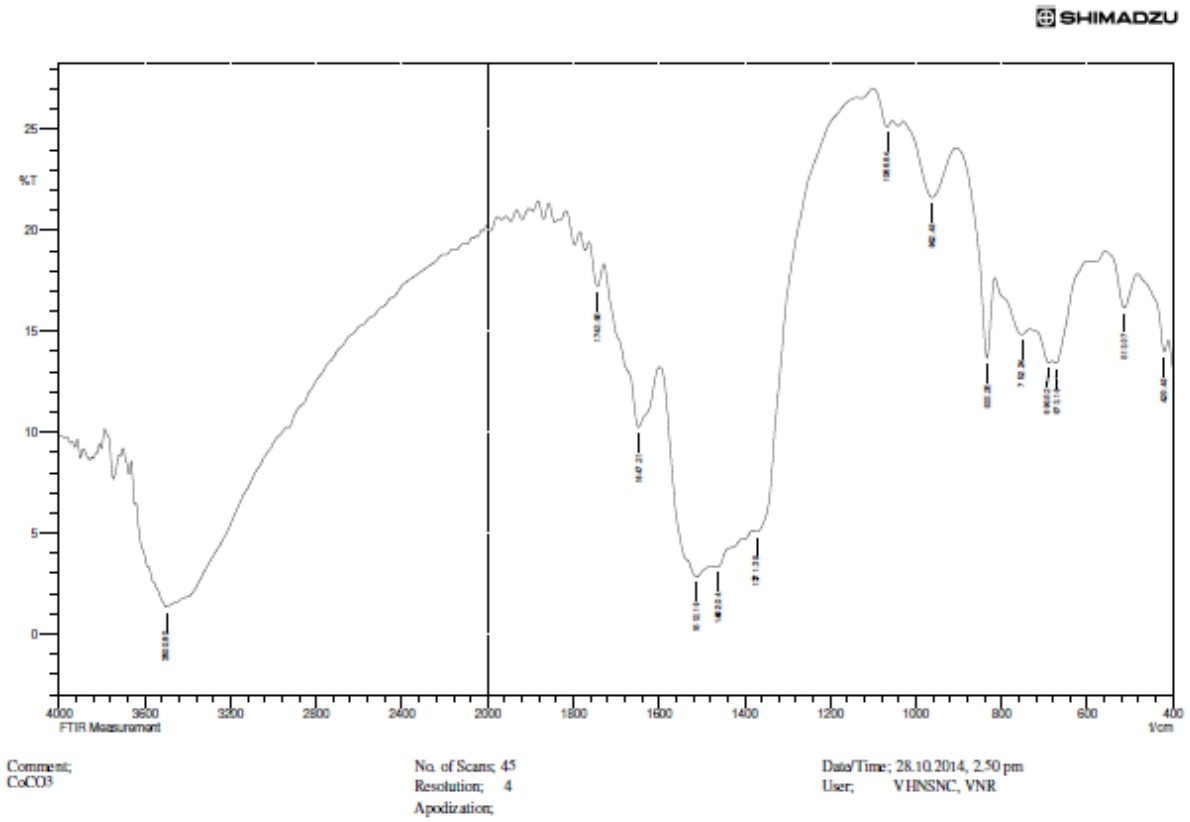

Figure.9 FTIR Spectra of Cobalt Carbonate Nanoparticles

4.4. UV Studies

The band gap of the prepared sample cobalt carbonate was determined by using UV visible studies. From the UV spectrum the optical band gap of cobalt carbonate nanoparticles is 5eV. Figure.9 shows graph to find the band gap of cobalt carbonate nanoparticles.

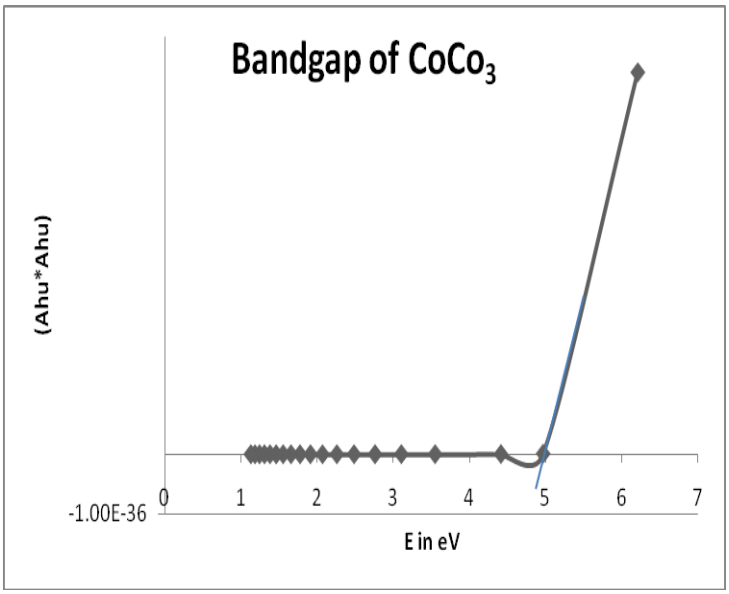

Figure.10 Graph to find the Band gap of
Cobalt Carbonate Nanoparticles

5. CONCLUSIONS

The **c**obalt carbonate nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (63.69 nm). 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.

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