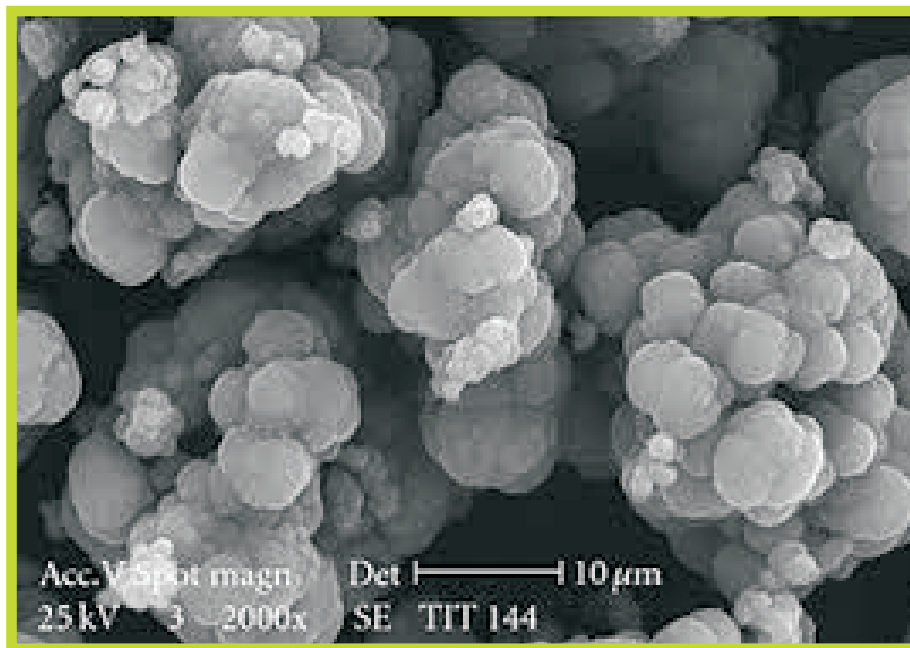


## REVIEW OF RESEARCH

### SYNTHESIS AND CHARACTERIZATION OF NICKEL CARBONATE NANOPARTICLES



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#### Abstract :

Nickel carbonate nanoparticles were synthesized via chemical co-precipitation method from nickel chloride and sodium carbonate. Structural and compositional properties were characterized by XRD, SEM, FTIR and UV spectroscopy X-ray diffraction (XRD) confirmed the preferential growth of nickel carbonate nanoparticles that width is 48.72nm. The SEM image shows the synthesized nickel 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 nickel carbonate nanoparticles is found to be 5eV.

**Keywords:** XRD, SEM, FTIR, UV.

## Synthesis And Characterization Of Nickel Carbonate Nanoparticles

### INTRODUCTION

Since the innovation of nanochemistry in past decades, numbers of materials in nanoscale have been synthesized via many methods. Nanomaterials have been widely used in various fields, such as photoelectric, recording materials, catalysts, sensors, ceramic materials, etc., due to their special structures and properties [1]. In particular Nickel carbonate is used to prepare nickel catalysts and several specialty compounds of nickel. It also is used as a neutralizing agent in nickel plating solutions. Other applications are in coloring glass and in the manufacture of ceramic pigments. These applications can be enhanced by decreasing the particle size and are highly dependent on particle size; the precise control of the size and distribution in a nanometer region is required. This work reports the synthesis of nickel carbonate nanoparticles by chemical co-precipitation method using nickel chloride and sodium carbonate and their characterization.

### 2. Experimental Details

Nanoparticles of nickel carbonate were prepared by chemical co-precipitation method by adding nickel 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 nickel carbonate nanoparticles 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 nickel carbonate nanoparticles are shown in fig.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 nickel carbonate nanoparticles are calculated using Scherrer equation

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

where  $\lambda$  represents wavelength of X rays,  $\beta$  represents half width at full maximum and  $\theta$  is the diffraction angle.

The average grain size of the particles is found to be 48.72nm. The peak list in the XRD pattern is given in table-1.

**Table-1. Intensity of XRD peaks.**

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
27.42	40	0.12	3.25046	14.35
31.732	276	0.17	2.81763	100.00
45.479	154	0.22	1.99278	55.65
56.44	31	0.37	1.62902	11.13

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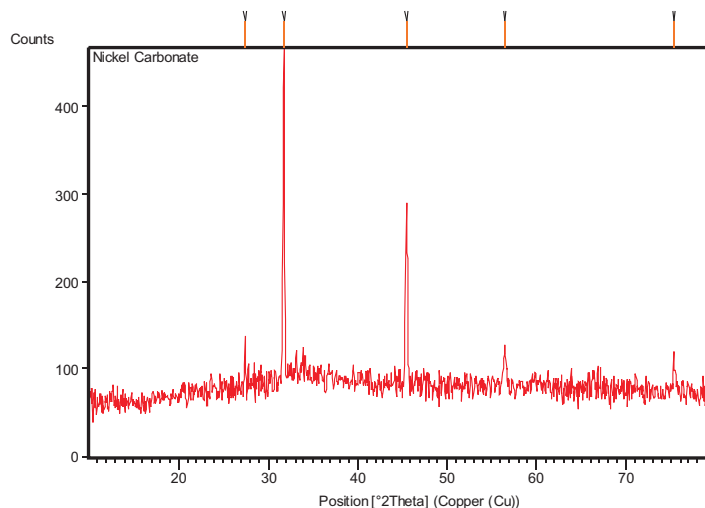


Fig.1 XRD pattern of nickel carbonate nanoparticles.

A good agreement between the Experimental diffraction angle  $[2\theta]$  and Standard diffraction angle  $[2\theta]$  of specimen is confirming standard of the specimen. Four peaks at  $2\theta$  values of nickel carbonate is observed and tabulated in table-2 and compared with the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), nickel carbonate file No. 12-0276. The d-spacing values of experimental is also confirming to the standard values.

**Table.2. Experimental and standard diffraction angles of nickel carbonate specimen.**

Experimental		Standard – JCPDS 12-0276	
Diffraction angle ( $2\theta$ in degrees)	D spacing ( $\text{\AA}$ )	Diffraction angle ( $2\theta$ in degrees)	D spacing ( $\text{\AA}$ )
27.42	3.25046	27.7668	3.21
31.732	2.81763	31.473	2.84
45.479	1.99278	45.64	1.986
56.44	1.62902	56.249	1.6340

### 4.1.2. XRD - Expected $2\theta$ Positions

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

Bragg's Law:  $2d \sin \theta = n\lambda$

$$d = \frac{\lambda}{2 \sin \theta} \quad (n = 1)$$

Wavelength  $\lambda = 1.5418 \text{ \AA}$  for  $\text{Cu K}\alpha$

The expected  $2\theta$  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-2.

$$1/d^2 = 1/\sin^2\beta((h^2/a^2)+(k^2\sin^2\beta/b^2)+(l^2/c^2) - (2hl\cos\beta/ac))$$

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Bragg's Law is used to determine the  $2\theta$  value: The expected  $2\theta$  and  $d$  values are close with the experimental  $2\theta$  and  $d$  values [2].

**Table 2. The Lattice plane and the lattice spacing from  $d$  from XRD of nickel carbonate nanoparticles.**

hkl	$2\theta$ (deg)		$d$ (Å)	
	Experiment	Expected	Experiment	Expected
310	27.42	27.712	3.25046	3.2151
222	31.732	30.729	2.81763	2.9061
512	45.479	44.315	1.99278	2.0416
604	56.44	54.1578	1.62902	1.6915

### 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 ( $d$ ) in the sample has been determined using expression

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

Where  $d$  is dislocation density and  $D$  is the crystallite size. Results of the dislocation density calculated from the formula is given in table-3. The number of unit cell is calculated from

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

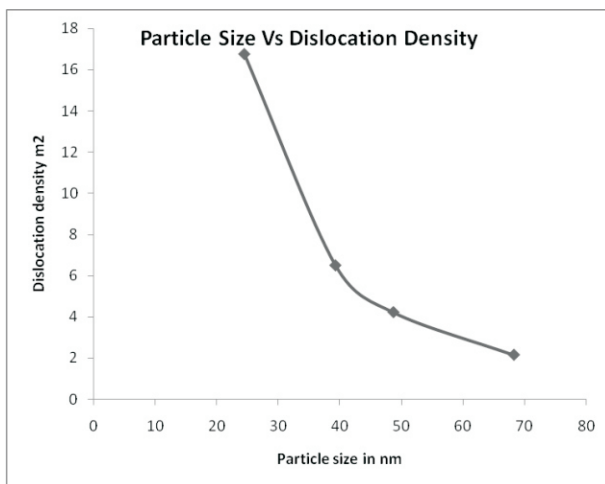
Where  $D$  is the crystallite size and  $V$  is the cell volume of the sample [3].

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

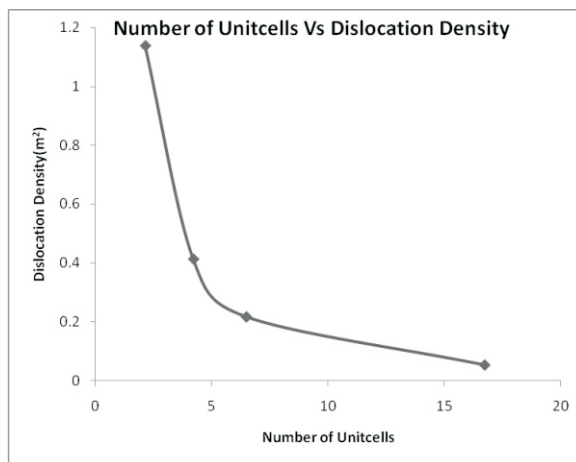
$2\theta$ (deg)	Particle Size $D$ (nm)	Dislocation Density ( $m^{-2}$ ) $\delta = 1 / D^2$ $\times 10^{14}$	Number of Unit Cell $\times 10^{05}$
27.42	68.3260	27.42	68.3260
31.732	48.7117	31.732	48.7117
45.479	39.2583	45.479	39.2583
56.44	24.4325	56.44	24.4325

## Synthesis And Characterization Of Nickel Carbonate Nanoparticles

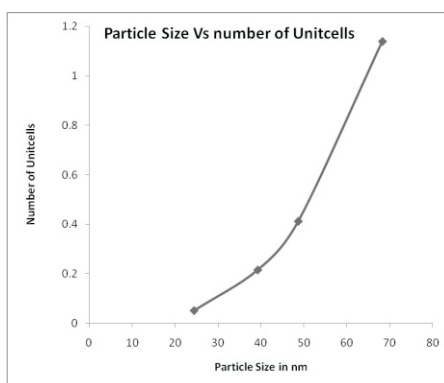
It is observed from these tabulated details, and from fig.2, fig.3 and fig.4, dislocation density is indirectly proportional to particle size and number of unit cells. Dislocation density increases while both particle size and number of unit cell decreases.



**Fig.2 Particle size Vs dislocation density for nickel carbonate nanoparticles.**



**Fig.3 Number of Unit cells Vs dislocation density for nickel carbonate nanoparticles.**



**Fig.4 Particle size Vs Number of unit cells for nickel carbonate nanoparticles.**

## Synthesis And Characterization Of Nickel Carbonate Nanoparticles

### 4.1.4. XRD – Morphology Index

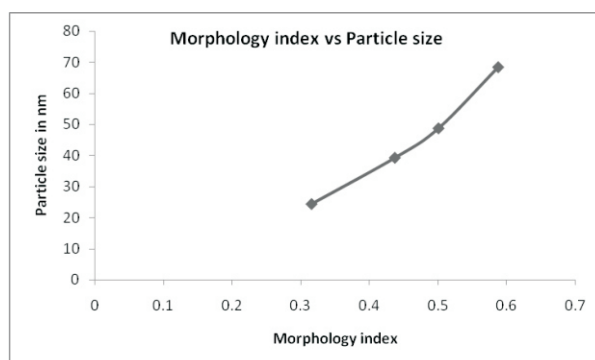
A XRD morphology index (MI) is calculated from FWHM of XRD data using the relation

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

Where M.I. is morphology index, FWHM<sub>h</sub> is highest FWHM value obtained from peaks and FWHM<sub>p</sub> 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)
0.00208	68.3260	0.5868
0.00296	48.7117	0.5
0.00382	39.2583	0.4365
0.00643	24.4325	0.3153



**Fig.5 Morphology Index of nickel carbonate nanoparticles.**

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

### 4.1.5. XRD – Unit Cell Parameters

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

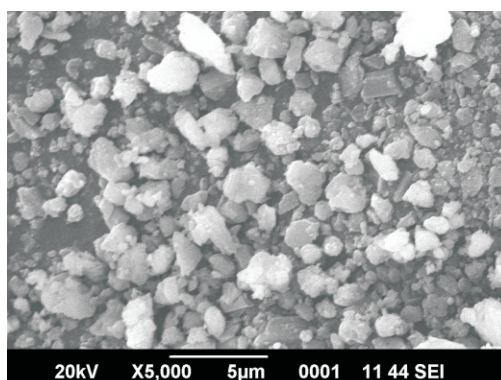
## Synthesis And Characterization Of Nickel Carbonate Nanoparticles

**Table-6. XRD parameters of nickel carbonate nanoparticles.**

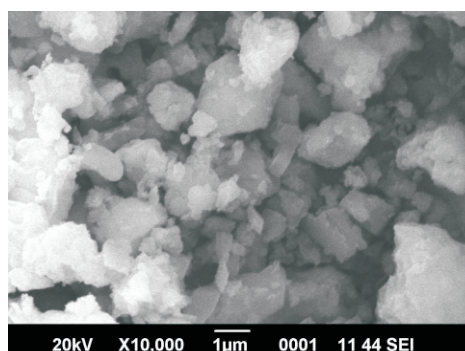
Parameters	Values
Structure	Monoclinic
Space group	C2/C[15]
Symmetry of lattice	End-centered
Particle size	48.72nm
Lattice parameters	a=10.77;b=7.299;c=18.681
Vol.unit cell(V)	1464.94
Density	2.057
Dislocation Density	$31.73 \times 10^{14}$
Mass	226.8amu

### 4.2. SEM studies

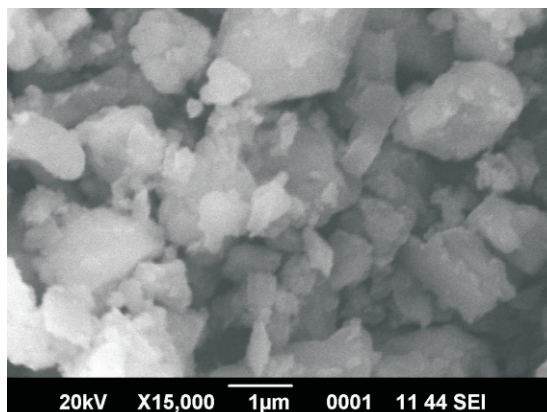
Scanning electron microscopy was used to analyze the morphology and size of the synthesized nickel carbonate nanoparticles. fig.2, fig.3, fig.4 and fig.5 show the SEM images of the nickel carbonate nanoparticles at various magnifications. The SEM images of nickel carbonate nanoparticles show well crystallized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



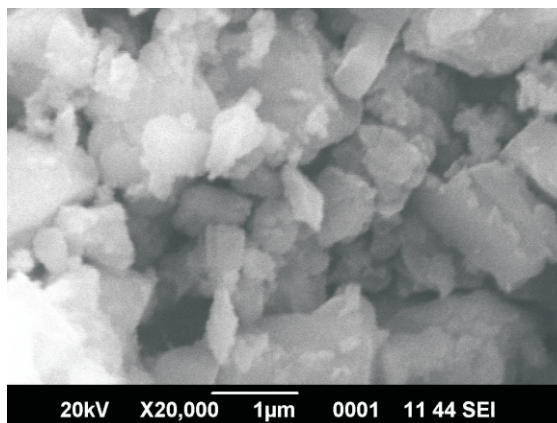
**Fig.2 SEM image at 5000 magnifications.**



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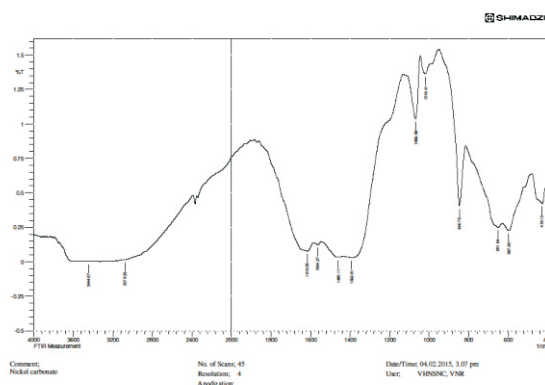


**Fig.3 SEM image at 10000 magnifications.**



### 4.3. FTIR Studies

The FTIR spectrum of the nickel carbonate sample is shown in the fig.6. The FTIR spectrum for nickel carbonate shows strong peaks at  $3444.87\text{ cm}^{-1}$  and  $3078.39\text{ cm}^{-1}$  corresponding to the free O-H group [3]. Another peak with a maximum of  $1616.35\text{ cm}^{-1}$  and  $1564.27\text{ cm}^{-1}$  are due to the bending mode of the hydroxyl group of water [3]. The spectrum also shows peak at  $846.75\text{ cm}^{-1}$  is due to carbonate ions [4] and  $430.13\text{ cm}^{-1}$  is due to the presence of Ni-O [5].



**Figure.9 FTIR spectra of nickel carbonate nanoparticles.**

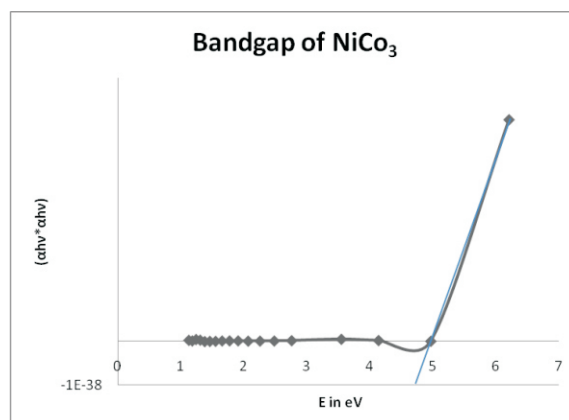
### 4.4. UV Studies

The band gap of the prepared sample nickel carbonate was determined by using UV visible studies. From the UV spectrum the optical band gap of nickel carbonate nanoparticles is  $5\text{ eV}$ . Fig. 10 shows the graph to find the band gap



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of nickel carbonate nanoparticles.



**Fig.10 Graph to find the band gap of nickel carbonate nano particles.**

The nickel carbonate nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range. 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.

### 6. REFERENCES

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