SYNTHESIS AND CHARACTERIZATION OF MAGNESIUM CARBONATE NANOPARTICLES

R.Hepzi Pramila Devamani*

R.Rajalakshmi**

M.Ranjani**

Abstract

Magnesium carbonate nanoparticles were synthesized via chemical co-precipitation method from magnesium sulfate 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 magnesium carbonate nanoparticles that width is 55.26nm. The SEM image shows the synthesized magnesium 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 magnesium carbonate nanoparticles is found to be 5eV.

Keywords: XRD, SEM, FTIR, UV.

* Assistant Professor, Post Graduate Department of Physics, V.V.Vanniaperumal College for Women, Virudhunagar.

** M.Sc Students, Post Graduate Department of Physics, V.V.Vanniaperumal College for Women, Virudhunagar.

1. Introduction

Magnesium carbonate occurs in nature in several minerals as hydrated, basic and double salts, as shown above. The two principal minerals are magnesite, $MgCO_3$ and dolomite, a double salt, $CaCO_3 \cdot MgCO_3$. Both minerals are used as source materials in the production of magnesium metal. Also, they are calcined to produce basic refractory bricks. Other applications of magnesium carbonate are in flooring, fireproofing and fire-extinguishing compositions; as a filler material and smoke suppressant in plastics; as a reinforcing agent in neoprene rubber; as a drying agent and for color retention in foods; in cosmetics; in dusting powder; a laxative to loosen the bowels; color retention in foods and in toothpaste. The high purity magnesium carbonate is used as an antacid in medicine; and as an additive to table salt. Another important application of magnesium carbonate is as a starting material in producing a number of magnesium compounds [1]. Magnesium carbonate, most often referred to as 'chalk', is used as a drying agent for hands in rock climbing, gymnastics, and weight lifting. Magnesium carbonate is also used in taxidermy for whitening skulls. It can be mixed with hydrogen peroxide to create a paste, which is then spread on the skull to give it a white finish.

2 .Experimental Details

Nanoparticles of magnesium carbonate were prepared by chemical co-precipitation method by adding magnesium sulfate 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 magnesium 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 magnesium carbonate nanoparticles 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.

The average grain size of the particles is found to be 55.26nm. The peak list in the XRD pattern is given in table-1.

Table-1.Intensity of XRD peaks.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
15.28	29	0.16	5.79492	13.89
19.023	100	0.12	4.66165	47.08
23.15	22	0.3	3.83873	10.27
28.019	90	0.16	3.18192	42.74
28.963	61	0.15	3.08033	28.91

30.78	37	0.37	2.90264	17.73
32.107	211	0.15	2.78551	100.00
33.833	152	0.12	2.64725	71.95
38.593	63	0.13	2.33100	29.71
40.78	25	0.18	2.21108	11.87
41.81	20	0.33	2.15865	9.27
45.41	8	0.4	1.99571	3.97
48.785	68	0.23	1.86519	32.28
49.44	13	0.2	1.84206	6.35
52.7	3	0.2	1.73642	1.35
54.58	22	0.18	1.68017	10.63
59.44	26	0.3	1.55375	12.30
61.6	5	1.0	1.50467	2.53
65.22	22	0.12	1.42936	10.20
71.00	7	0.7	1.32654	3.49
72.93	16	0.18	1.29614	7.34
74.00	9	0.4	1.27994	4.20

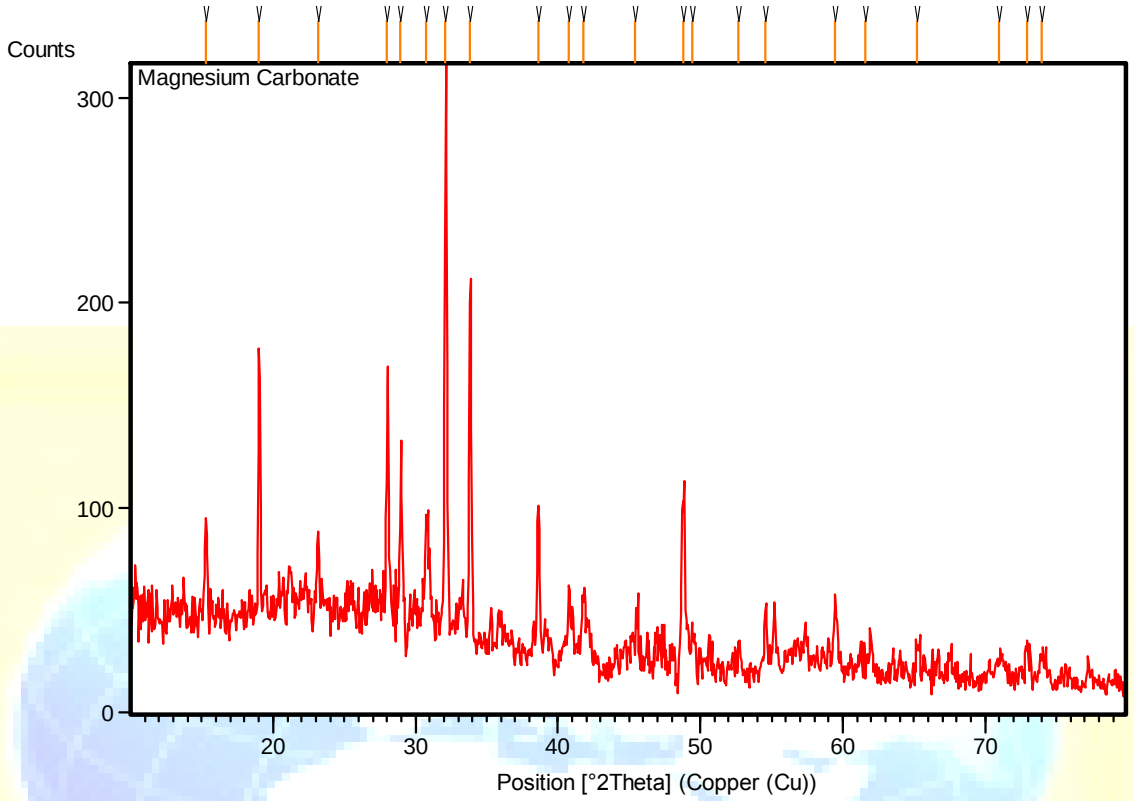

Fig.1 XRD pattern of magnesium carbonate nanoparticles.

A good agreement between the Experimental diffraction angle [2θ] and Standard diffraction angle [2θ] of specimen is confirming standard of the specimen. Four peaks at 2θ 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. 80-0101. The d-spacing values of experimental is also confirming to the standard values.

Table.2. Experimental and standard diffraction angles of magnesium carbonate specimen.

Experimental		Standard – JCPDS 80-0101	
Diffraction angle (2θ in degrees)	D spacing (Å)	Diffraction angle (2θ in degrees)	D spacing (Å)
32.107	2.78551	32.586	2.7456

38.593	2.33100	38.985	2.3085
52.7	1.73642	51.691	1.7669
54.58	1.68017	53.833	1.7016
61.6	1.50467	61.628	1.5037
71.00	1.32654	70.613	1.3328

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.

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

Where δ 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.

2θ (deg)	Particle Size D (nm)	Dislocation Density (m^2) $\delta = 1 / D^2$	Number of Unit Cell $X10^{05}$

		$X10^{14}$	
32.107	55.26	3.2749	3.1659
38.593	64.92	2.3727	5.1339
52.7	44.45	5.0612	1.6478
54.58	49.80	4.03219	2.3174
61.6	9.27	1.1628	1.4963
71.00	13.97	5.1188	5.1234

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.

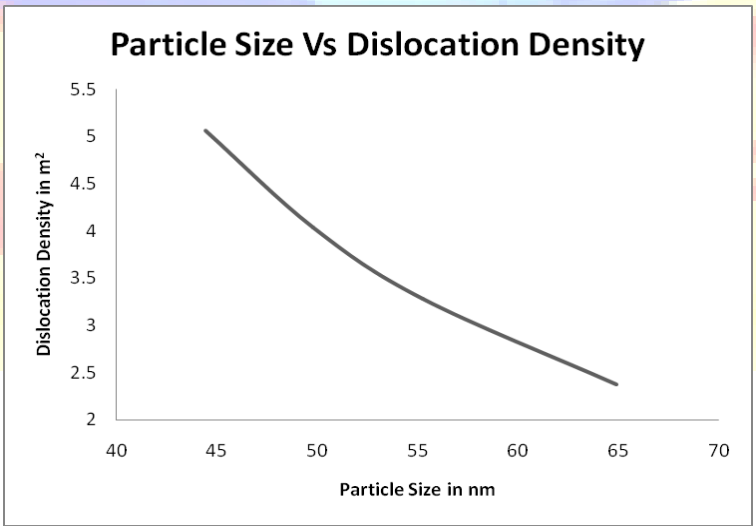

Fig.2 Particle size Vs dislocation density for magnesium carbonate nanoparticles.

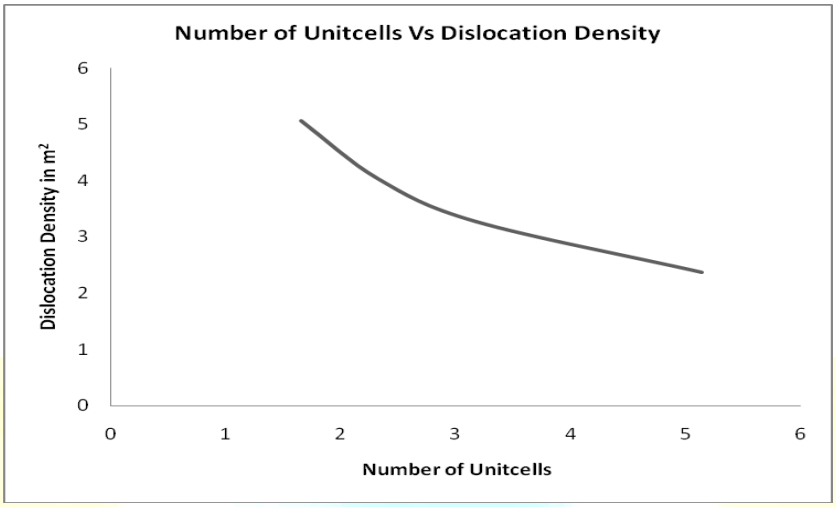

Fig.3 Number of Unit cells Vs dislocation density for magnesium carbonate nanoparticles.

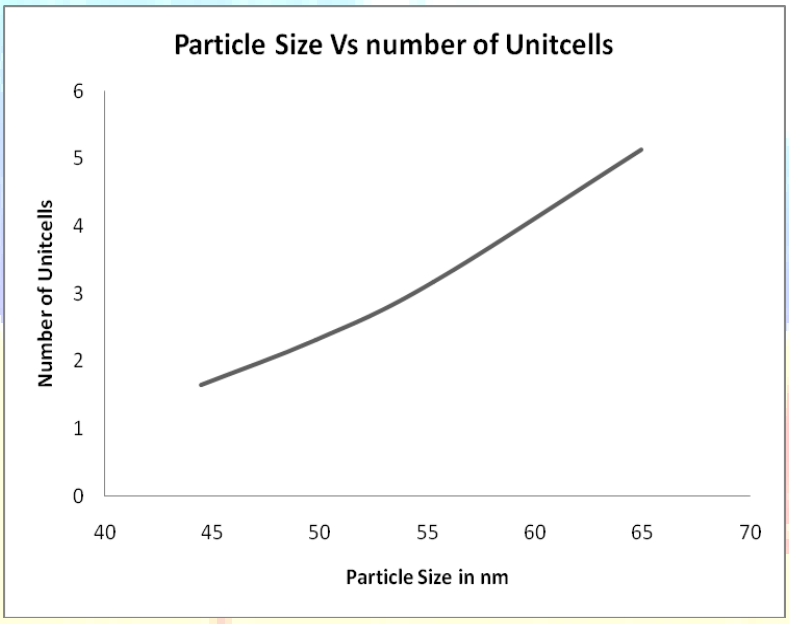

Fig.4 Particle size Vs Number of unit cells for magnesium carbonate nanoparticles.

4.1.4. XRD – Morphology Index

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

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

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-4.

Table-4. Relation between Morphology Index and Particle size.

FWHM (β) radians	Particle Size(D) nm	Morphology Index (unitless)
0.002616	55.26	0.5
0.002267	64.92	0.5357
0.003488	44.45	0.4286
0.003139	49.80	0.4546
0.0174	9.27	0.1307
0.012208	13.97	0.1765

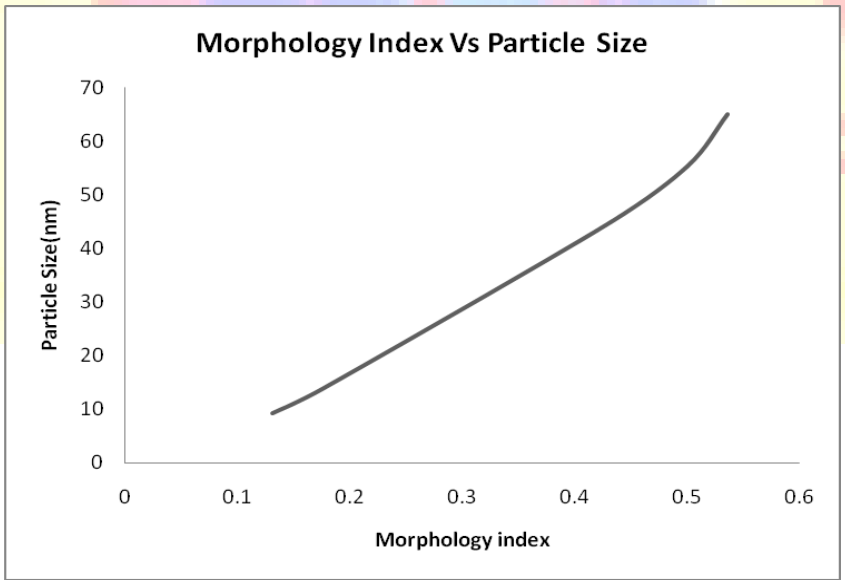

Fig.5 Morphology Index of magnesium carbonate nanoparticles.

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

Table-6. XRD parameters of nickel carbonate nanoparticles.

Parameters	Values
Structure	Rhombohedral
Space group	R3C[167]
Symmetry of lattice	Rhomb-centered
Particle size	55.26nm
Lattice parameters	A=4.617;c=15.108
Vol.unit cell(V)	278.91
Density	3.012
Dislocation Density	$3.2749\ X10^{14}$
Mass	84.31amu

4.2. SEM studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized magnesium carbonate nanoparticles. fig.2, fig.3, fig.4 and fig.5 show the SEM images of the magnesium carbonate nanoparticles at various magnifications. The SEM images of magnesium 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.

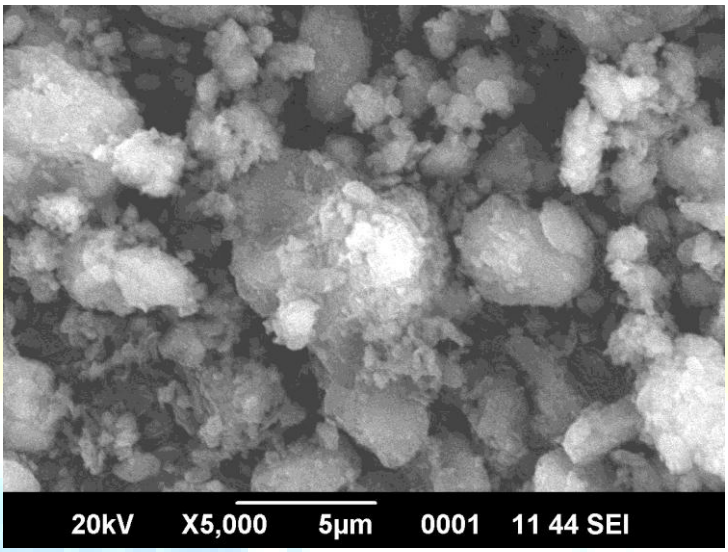

Fig.2 SEM image at 5000 magnifications.

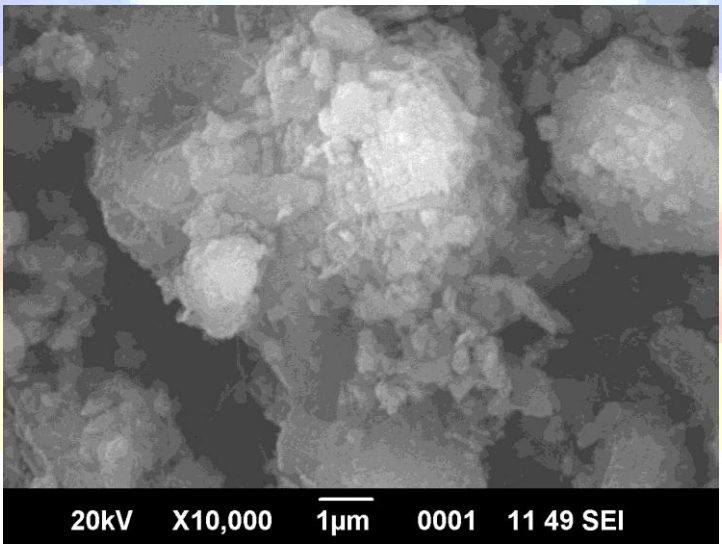

Fig.3 SEM image at 10000 magnifications.

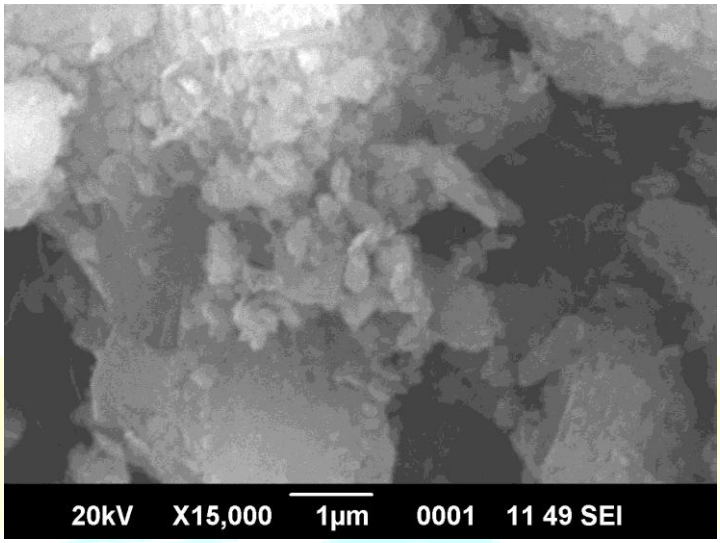

Fig.4 SEM image at 15000 magnifications.

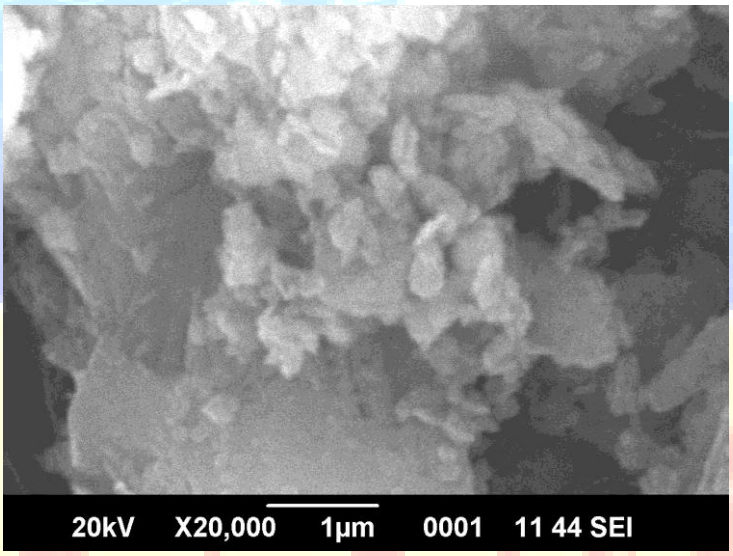

Fig.5 SEM image at 20000 magnifications.

4.3. FTIR Studies

The FTIR spectrum of the magnesium carbonate sample is shown in the fig.6.The FTIR spectrum for magnesium carbonate shows strong peaks at 3647.39 cm^{-1}, 3514.30 cm^{-1} and 3431.36 cm^{-1} corresponding to the free O-H group [2]. Another peak with a maximum of 1523.76 cm^{-1} are due to the bending mode of the hydroxyl group of water [2]. The spectrum also

show peaks at 883.40 cm^{-1} due to carbonate ions [3] and 592.15 cm^{-1}, 441.70 cm^{-1} are due to the presence of Mg-O vibrations [4].

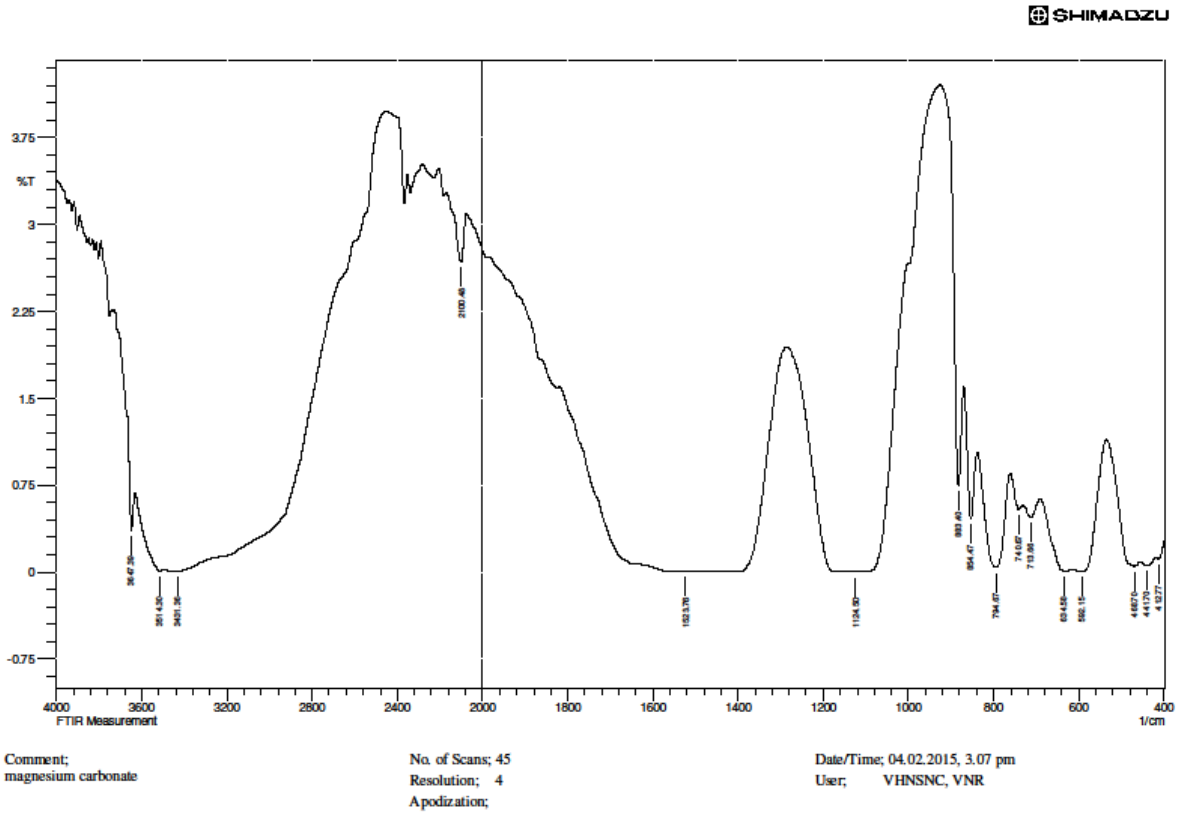

Figure.9 FTIR spectra of magnesium carbonate nanoparticles.

4.4. UV Studies

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

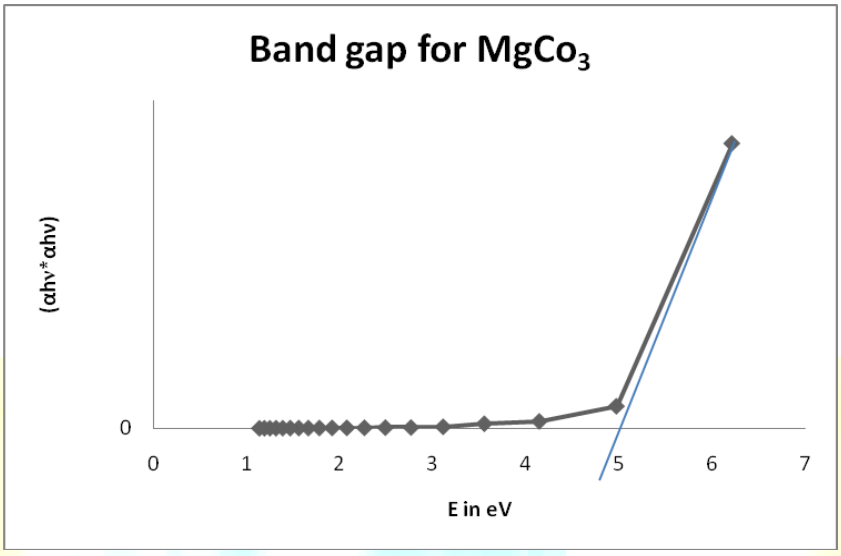

Fig.10 Graph to find the band gap of magnesium carbonate nano particles.

5. CONCLUSIONS

The magnesium 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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