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Synthesis and Characterization of Nanoparticles of Magnesium Oxide (MgO)

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Abstract

Composite-hydroxide-mediated (CHM) method is used to prepare magnesium oxide, in this preparation magnesium chloride (MgCl2) salt is used with specific ratios of Sodium hydroxide (NaOH) and Potassium hydroxide (KOH) and the whole reaction done in a vacuum less muffle furnace, firstly the product was obtained is magnesium hydroxide Mg(OH)2. We annealed this product at different temperatures to discuss the percentage composition of magnesium oxide in magnesium hydroxide and also discuss the temperature at which magnesium hydroxide is completely converted in magnesium oxide. For indirect prepared magnesium oxide we discussed, Grain size, Structure, Miller indices, Lattice constant, Strain, Dislocation density and Density using XRD technique and also discussed Grain size, Absorbance, Reflectance, Transmission, band gap and conductance using UV-visible technique and for accurate size estimation we used SEM technique.

Keywords: Composite-Mediated-Hydroxide (CHM); X-rays Diffraction (XRD); Ultraviolet-Visible (UV); Scanning Electron Microscope (SEM).

1. Introduction

1.1. Introduction to Nanotechnology

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The word nanotechnology was introduce for the first time into a scientific world by N.Taniguchi at the international conference on industrial production in Tokyo in 1974 in order to describe the super thin processing of materials with nanometer accuracy and the accuration of nanosized mechanisms.

In 1991 the first nanotechnological programme of National Scientific Fund started to operate in USA [1].

It can be defined as the design, synthesis and application of materials whose size and shape have been designed at nano scale. It exploits unique chemical physical electrical and mechanical properties that emerge when matter is structured at nano scale [2].

1.2. Introduction to XRD

The term XRD is derived from X-ray diffraction, which is used to study the characteristics of a crystalline material. Among all solid materials about 95% can be illustrated as crystalline. X-ray diffraction pattern of a pure crystalline material is just similar to a fingerprint of the material. Hence preferably, The X-ray diffraction technique is appropriate for classification and detection of polycrystalline phases. The most important benefit of powder diffraction is to classify components in a sample by a search/match formula. Moreover, the areas under the peak are associated with the quantity of each phase present within the test sample [3].

1.3. Introduction to UV-visible

The spectroscopy which makes use of both the ultraviolet (UV) and the visible range of electromagnetic radiation is normally known as UV-visible Spectroscopy. The term reveals that inside the molecule, somewhat high energy photons disturb the electron sharing. As a result, the molecular orbital explanation of the molecular electron distribution and its alteration during excitations is extremely helpful [4].

1.4. Introduction to SEM

Basically, SEM is a microscope that makes use of electrons to produce an image. In order to produce various signals at the surface of solid sample, the scanning electron microscope (SEM) uses a focused beam of high-energy electrons. Scanning electron microscopes have opened new areas of learning in the medical and physical sciences, ever since their development in 1950. It has facilitated the researchers and learners to observe a much larger variety of samples [5].

1.5. Introduction to preparatory techniques CHM

The term CHM stands for ‘Composite-hydroxide-mediated ‘approach for synthesizing Nanoparticles’. This is one of the best methods to prepare Nanoparticles, it is technically sound (Important), environmentally friendly method to create a wide range of important Nanoparticles.

CHM is based on the chemical reactions of materials in eutectic hydroxide melts (Which are formed at the lowest possible temperature) at~ 200℃ and ambient pressure in the absence of organic dispersants (liquid or gas

used to diffuse small particles in a medium) or capping reagents (which cover the surface).

Although the melting point of pure NaOH, KOH & LiOH are above 300℃.

NaOH	323℃
KOH	404℃
LiOH	477℃

But the eutectic points (the lowest possible temperature at which a substance melts) for some mixtures are.

Compounds	Compounds Ratio	Eutectic Point
NaOH\KOH	51.5:48.5	165℃
LiOH\KOH	0.31:0.69	225℃
NaOH\LiOH	0.71:0.29	220℃

Due to these specific ratios melting point decreases & one of the given ratio we used in CHM method (Commonly we use the ratio, NaOH\KOH→51.5:48.5→165℃).

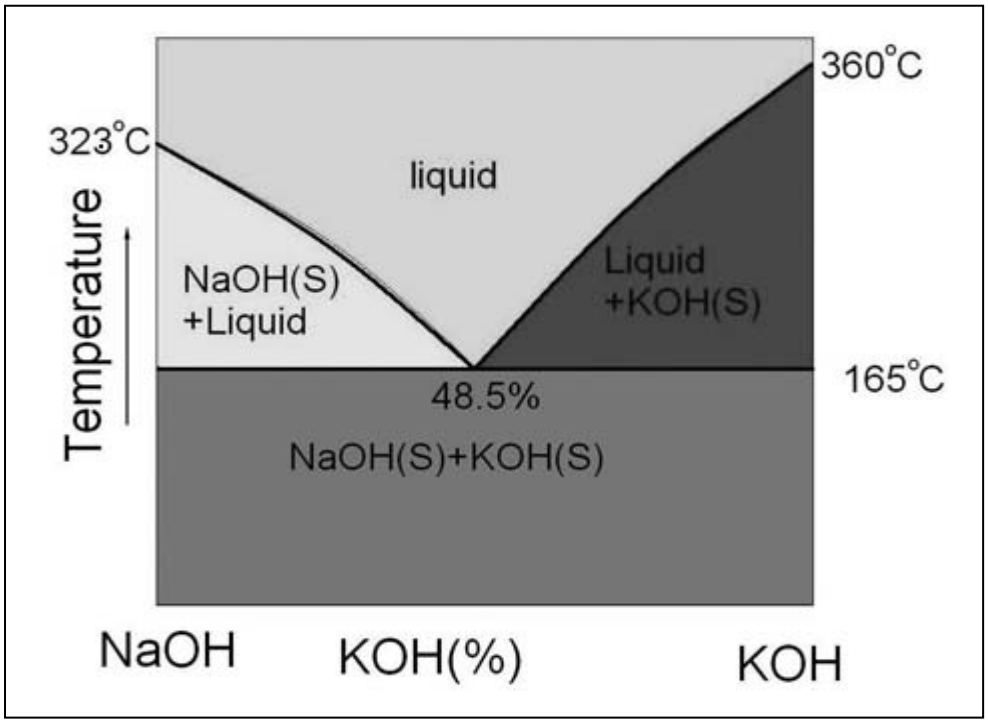


Figure 1: Eutectic point

Mixtures of these hydroxides are used as a medium during the reaction. All the raw materials with a certain amount of mixed hydroxide are placed within the Teflon vessel at the same time. Then, the nanostructures are

formed within the vessel after heating in a furnace at 200℃ for several hours or days [6].

2. Synthesis

First of all take pure sodium hydroxide (NaOH) 51.5% and potassium hydroxide (KOH) 48.5% in a Teflon beaker and then add the weighted amount of magnesium chloride (MgCl2) in it. Put the beaker in the furnace for about 5 minutes at 200℃, then take it out and shake the mixture well with the hand. After this again put the beaker in the furnace for 24hrs at 200℃.

Now after a reaction of 24hrs cool the mixture at room temperature and then wash it with distill water for 4-5 times to remove the residual yield which is produced during the reaction.

Table 1: Compounds Quantity.

Compound	Percentage Composition	Concentration in grams
NaOH	51.5%	4.635g
KOH	48.5%	4.365g
MgCl2	N/A	2g

Chemical Reaction

$$Mg(Cl)_2\ (s) + NaOH(s) + KOH(s) \xrightarrow[200℃\backslash 24hrs]{} Mg(OH)_2(s) + KCl\ (l) + NaCl\ (l)$$

$$Mg(OH)_2(s) \xrightarrow[210-630℃\backslash 18hrs]{} MgO(s) + H_2O(g)$$

Table 2: Compound Purity**.**

Compound	Purity
NaOH	95%
KOH	95%
MgCl2	90%

3. Characterization Studies

3.1. XRD Analysis

1) Discussion on Annealing Temperature

Annealing temperature is one of the important issues in CHM to convert hydroxides into oxides; we performed

annealing of $Mg(OH)_2$ to convert it in MgO at different temperatures. We found the range of annealing temperature which was not explained yet in CHM method.

Flow Sheet

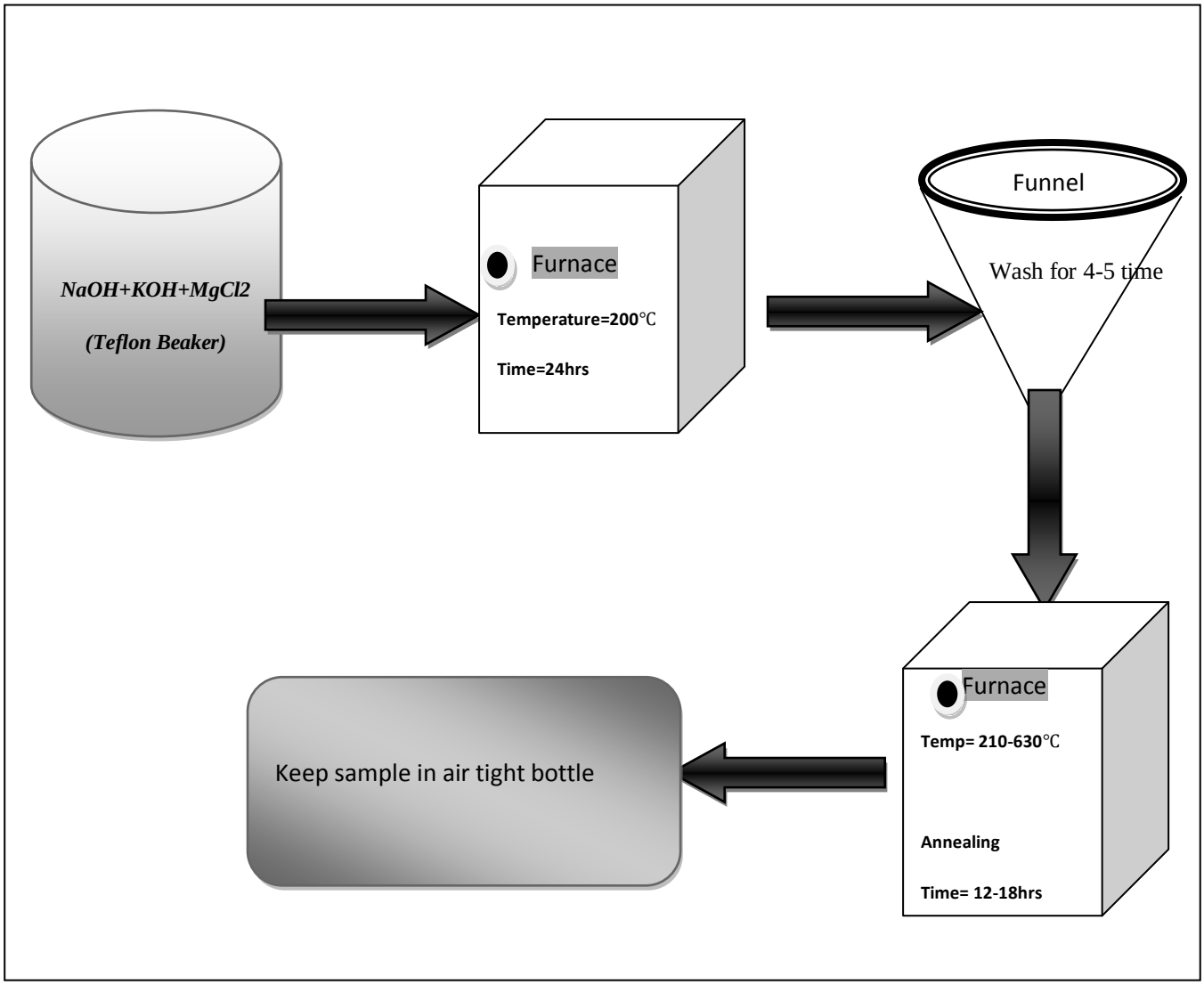


Figure2: Flow sheet

Table 3 Percentage Quantity

Sample	Annealing Temperature Range	Annealing Time	Resultants
Mg(OH)2	Room	till dry	Mg(OH)2**(60%)**/MgO**(40%)**
Mg(OH)2	25-70℃	2 hours	Mg(OH)2**(56%**) /MgO**(44%**)
Mg(OH)2	70-200℃	18hours	Mg(OH)2**(43%)**/MgO**(57%)**
Mg(OH)2	210-630℃	18hours	MgO**(83%)**

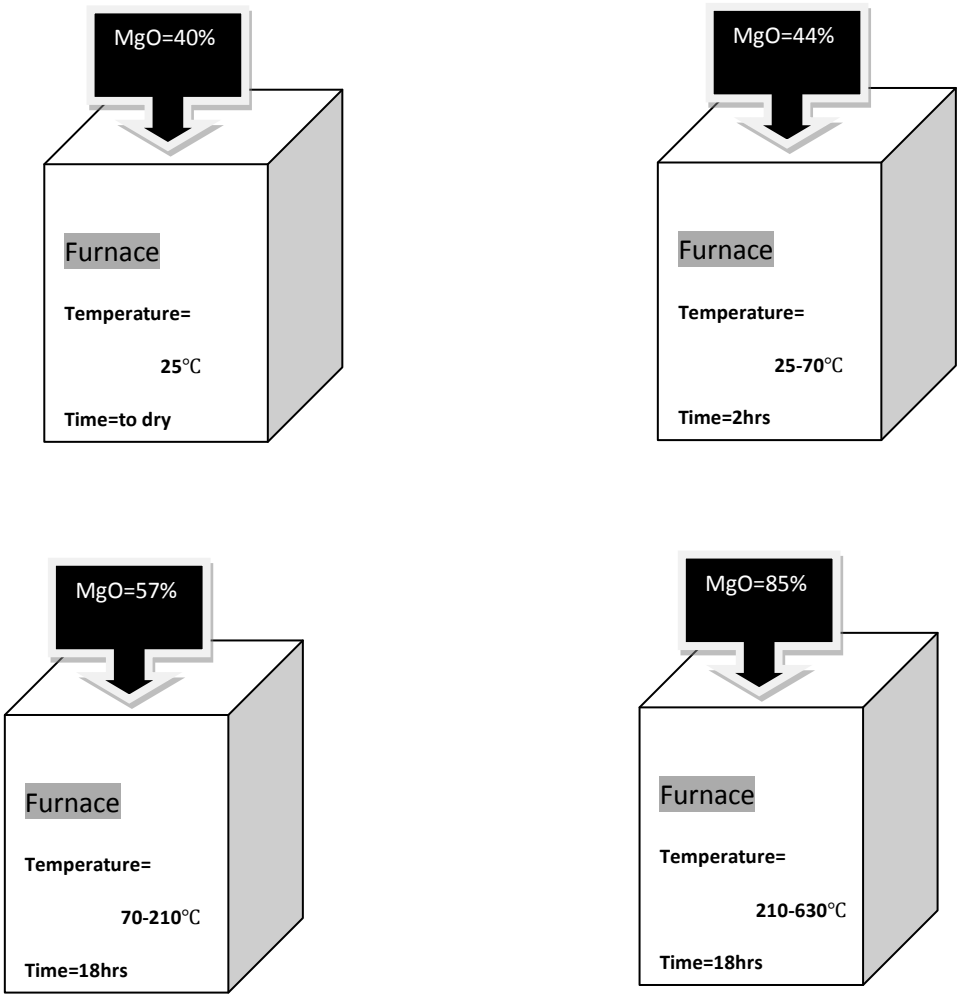


Figure 3: Annealing Time and Temperature

We are the first those explained the annealing temperature to convert the hydroxides into oxides in CHM method.

XRD Results

- ***Room Temperature (MgO 40%)***

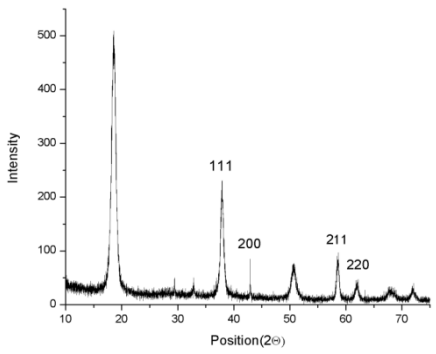


Table 4: MgO 40%

Total Peaks	Required Peaks	Obtained % of MgO	Required Angles	Required Planes
10	4	40	37.84	111
			42.82	200
			58.59	211
			61.93	220

❖ ***25-70℃(MgO 44%)***

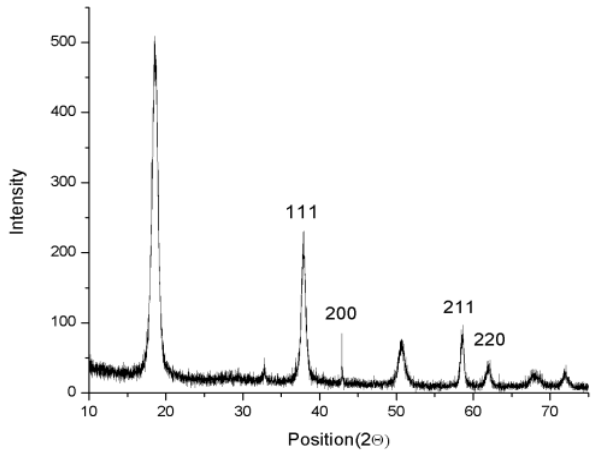


Table 5: MgO 44%

Total Peaks	Required Peaks	Obtained % of MgO	Required Angles	Required Planes
9	4	44.44	37.85	111
			42.83	200
			58.59	211
			61.93	220

❖ ***70-210℃(MgO 57%)***

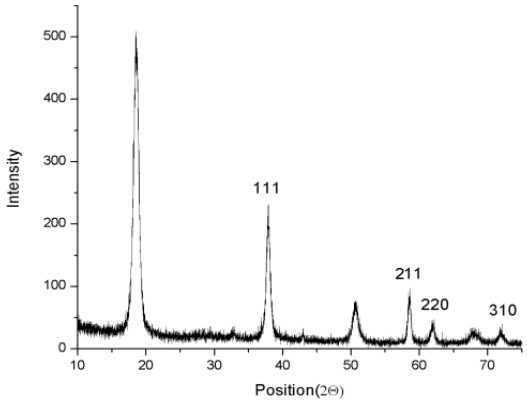


Table 6: MgO 57%

Total Peaks	Required Peaks	Obtained % of MgO	Required Angles	Required Planes
7	4	57.14	37.86	111
			58.60	211
			61.95	220
			72.14	310

❖ ***210-630℃(MgO 83%)***

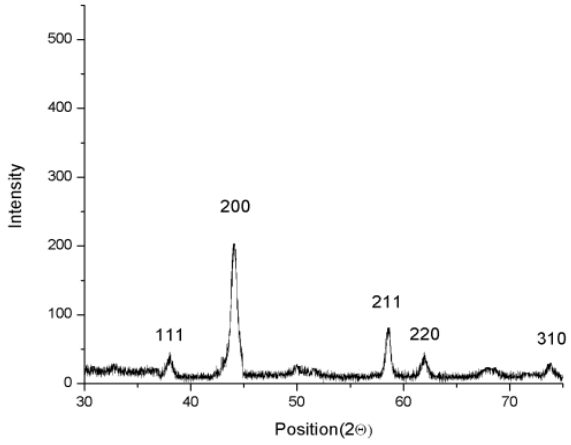


Table 7: MgO 83%

Total Peaks	Required Peaks	Obtained % of MgO	Required Angles	Required Planes
6	5	83.33	37.96	111
			43.93	200
			58.65	211
			62.10	220
			73.98	310

We obtained our product known as magnesium oxide at about 450℃ by annealing process, but the question is that why we annealed our sample using CHM method, because we used vacuum less furnace instead of vacuum furnace that's why the moisture in air is absorbed in our sample and produce magnesium hydroxide rather than magnesium oxide, because second group elements are highly unstable. Now to obtain magnesium oxide we anneal our first product at different temperatures but we observed there is a little change at low temperatures because moisture is absorbed in the inner layers of our sample not on the surface, but when we annealed our sample at about 450℃ we obtained pure magnesium oxide.

Modification in CHM

1) Vacuum-less furnace instead of vacuum furnace

2) Room pressure instead of desired pressure

3) Control system, manual instead of automatic

Shifting in Peaks

Table 8: Shifting in Peaks

Angles obtained with annealing for MgO	Standard Angles for MgO
37.96	38.43
43.93	44.37
58.65	59.17
62.10	64.52
73.98	73.33

This slightly shift in angles w.r.t the standard angles for MgO is because of the presence of stabilizing aluminum impurities.

2) Discussion with XRD Result

Using XRD result of magnesium oxide (MgO) we found.

- Grain Size
- Structure
- Miller Indices
- Lattice Constant
- Strain
- Dislocation Density
- Density

❖ ***Grain Size***

Derived expression for grain size is given as.

$$t=\frac{K\lambda}{BCos\theta_B}\text{.....(1)}$$

Where K=0.9

t=Grain size

λ=Wave length

B=Full width at half maximum (FWHM)

θ_B=Average angle

We know that

$$B=\frac{1}{2}(2\theta_2-2\theta_1)\text{..... (2)}$$

$$2\theta_B=(\frac{2\theta_1+2\theta_2}{2})\text{.....(3)}$$

Table 9: FWHM

Peak No	Angle(2θ)	$2\theta_1$	$2\theta_2$	(FWHM) B=$(2\theta_2-2\theta_1)/2$
1	37.96	37.528	38.438	0.455
2	43.93	43.706	44.486	0.390
3	58.65	58.173	58.953	0.390
4	62.10	61.512	62.422	0.455
5	73.98	73.380	74.180	0.400

Now put all values in equation (1) & (3)

Table 10: Grain Size

Peak No	Plane	X-ray Wave Length(λ)	FWHM(B) Rad	θ_1	θ_2	Average Angle(θ_B)	Cosθ_B	Grain Size(t)
1	111	1.54$\dot{A}$	7.937x10^{-3}	18.764	19.219	18.991	0.9455	18.46nm
2	200	1.54$\dot{A}$	6.803x10^{-3}	21.853	22.243	22.048	0.9268	21.98nm
3	211	1.54$\dot{A}$	6.803x10^{-3}	29.086	29.476	29.281	0.8722	23.35nm
4	220	1.54$\dot{A}$	7.937x10^{-3}	30.756	31.211	30.983	0.8573	20.36nm
5	310	1.54$\dot{A}$	6.977x10^{-3}	36.690	37.090	36.890	0.7997	24.84nm

The Average Grain size=21.79nm

❖ ***Miller indices***

We can find miller indices in five steps

- Identify the peaks & their proper 2θ values.
- Determine$Sin^2\theta$.
- Calculate ratio $\frac{Sin^2\theta}{Sin^2\theta_{(\min)}}$& multiply by the appropriate integer until the fraction vanishes.
- Write yield in terms of $h^2 + k^2 + l^2$.
- Find miller indices from the square term.

Table 11: hkl

Peak No	2θ	$Sin^2\theta$	$1x\frac{Sin^2\theta}{Sin^2\theta_{(\min)}}$	$2x\frac{Sin^2\theta}{Sin^2\theta_{(\min)}}$	$3x\frac{Sin^2\theta}{Sin^2\theta_{(\min)}}$	$h^2 + k^2 + l^2$	hkl
1	37.96	0.106	1	2	3	1+1+1	111
2	43.93	0.139	1.31	2.62	4	2+0+0	200
3	58.65	0.239	2.25	4.50	6	2+1+1	211
4	62.10	0.266	2.51	5.02	8	2+2+0	220
5	73.98	0.362	3.41	6.83	10	3+1+0	310

❖ ***Structure***

There are some conditions for cubic lattice.

Primitive: All possible h,k & l values

$$h^2 + k^2 + l^2 = 1,2,3,4,5\ldots..$$

BCC: Reflection is allowed when (h+k+l) is even

Reflection not allowed when (h+k+l) is odd

$$h^2 + k^2 + l^2 = 2,4,6,8,10\ldots.$$

FCC: Reflection is allowed when h,k & l all are even or odd.

Reflection is not allowed when h,k & l are mixed, even or odd.

$$h^2 + k^2 + l^2 = 3,4,8,11,12,16,19\ldots..$$

This result shows that structure of pure magnesium oxide is cubic

❖ ***Lattice Constant***

We have an expression which tells us about the relation between the diffraction angle & the Miller indices, using this expression we can find lattice constant and the expression is given as.

Table 12: Structures

Hkl	$h^2 + k^2 + l^2$	Sc	BCC	FCC
111	3	√	X	√
200	4	√	√	√
211	6	√	√	X
220	8	√	√	√
310	10	√	√	X

$$Sin^2\theta \propto (h^2 + k^2 + l^2)$$

$$Sin^2\theta = \frac{\lambda^2}{4a^2}(h^2 + k^2 + l^2) \ldots\ldots\ldots\ldots (1)$$

$$a^2 = \frac{\lambda^2}{4Sin^2\theta}(h^2 + k^2 + l^2) \ldots\ldots\ldots\ldots (2)$$

Table 13: Lattice Constant

hkl	Diffraction Angle(2θ)	θ	X-ray Wave Length(λ)	Lattice Constant(a)$\dot{A}$
111	37.96	18.98	1.54$\dot{A}$	4.10
200	43.93	21.96	1.54$\dot{A}$	4.12
211	58.65	29.32	1.54$\dot{A}$	3.85
220	62.10	31.05	1.54$\dot{A}$	4.22
310	73.98	36.99	1.54$\dot{A}$	4.05

The average value of lattice constant=4. 15$\dot{A}$

❖ ***Strain***

In crystals strain may shift the peaks from the diffraction line, Macro strain may disturb the lattice parameters as a result peaks in XRD shifts from their original position. Micro strain can be produced by tensile and compressive forces which actually increase the FWHM or broadening the diffraction peaks in some cases. Dislocations, vacancies, shear planes can also produce Micro stress.

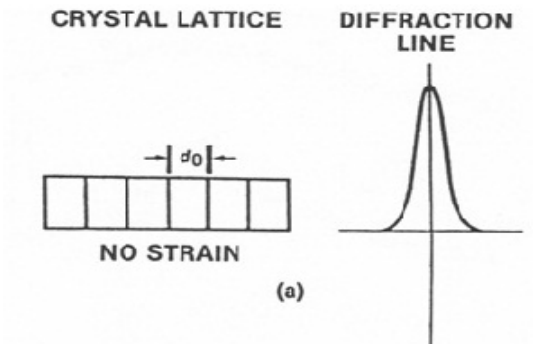


Figure 4: No Strain

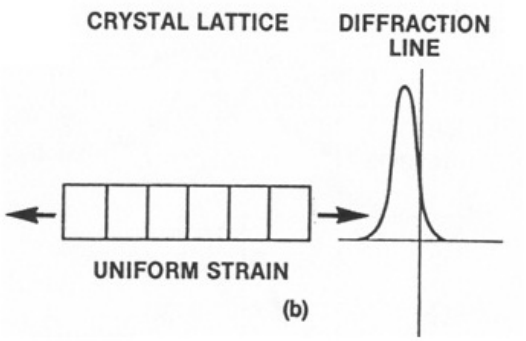


Figure 5: Uniform Strain

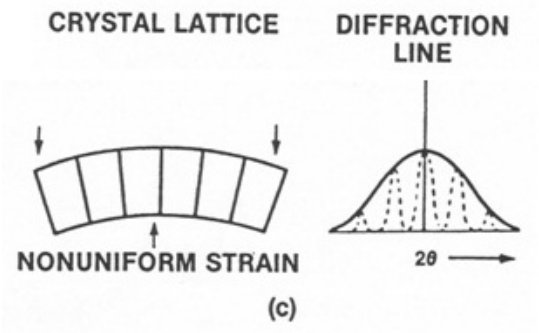


Figure 6: Non-Uniform Strain

COURTESY: Diffraction Basics Intensities, EPS400-002, Introduction to X-ray Powder Diffraction, Jim Connolly, spring 2012.

We found a strain for magnesium oxide Nanoparticles using the results of XRD, as

Strain=$\frac{\mathbf{BCos\theta_B}}{\mathbf{4}}$

Table 14: Strain

Planes	**FWHM(Rad)**	**Cosθ_B**	**Strain**
111	$7.937x10^{-3}$	0.9455	0.001876108
200	$6.803x10^{-3}$	0.9268	0.001576255
211	$6.803x10^{-3}$	0.8722	0.001483394
220	$7.937x10^{-3}$	0.8573	0.001701098
310	$6.977x10^{-3}$	0.7997	0.001394877

The average strain=0.0016064064

We observed our peaks for MgO are slightly displaced from their original position this is because of large strain as we calculated above. Calculated strain is also very small itself but large as compared to the standard strain for magnesium oxide.

❖ ***Dislocation Density***

We can find the dislocation density using a relation which is given as.

$\sigma=\frac{1}{d^2}$**……..….(1)**

Where
σ=Dislocation
d=t=Grain size

Table 15: Dislocation

Grain Size(d)	**d^2**	**Dislocation(σ)**
18.46	340.7716	0.002934516844
21.98	483.1204	0.002069877405
23.35	545.2225	0.001834113596
20.36	414.5296	0.002412372964
24.84	617.0256	0.001620678299

The average dislocation density=0.002174311822

❖ ***Density***

Lattice constant of magnesium oxide (MgO) =4.15$\dot{A}$=4.15x10^{-8}cm

Volume of magnesium oxide (MgO) =v=a^3=71.4733x$10^{-24}cm^3$

Mass of magnesium oxide (MgO) =40.29

Weight of one magnesium oxide (MgO) atom=40.29/6.022x10^{23}

Table 16: Density

Sample	$v = a^3$ (cm^3) **for cubic system**	**Weight of one atom for MgO**	**Weight of one unit cell for MgO (m)**	$\rho = \frac{m}{v}$ **(g.cm^{-3})**
MgO	71.4733x10^{-24}	66.90x10^{-24}	267.6x10^{-24}	3.74

Our calculated density is 0.2 times greater than the real density of magnesium oxide Nanoparticles this is also because of slightly shift in peaks position.

❖ ***Comparison of our results with standard results***

Table 17: Comparison

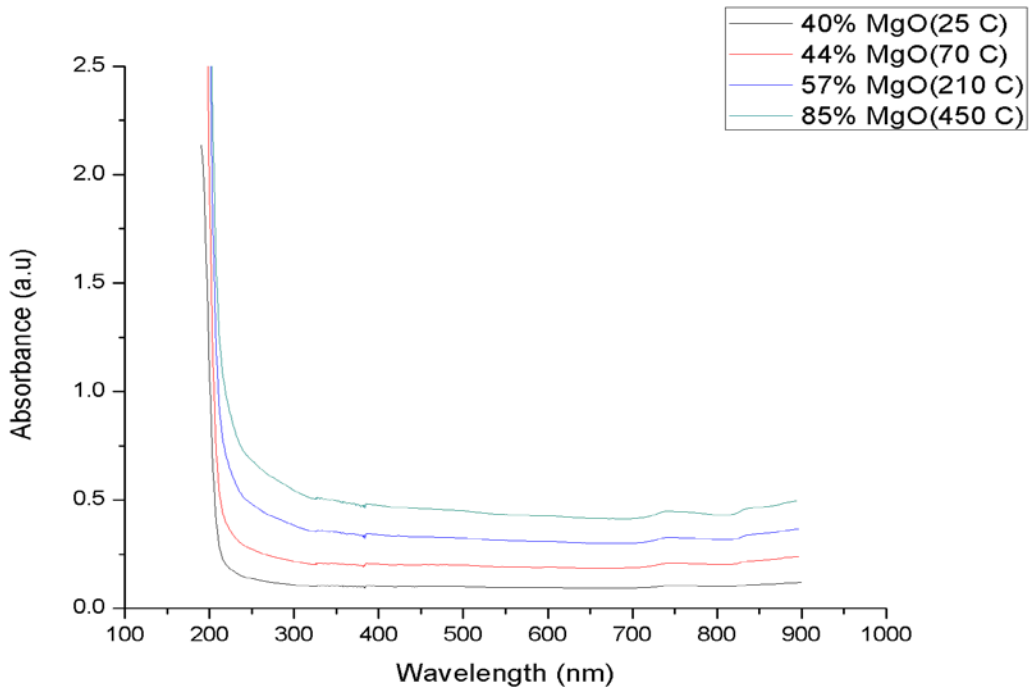
Findings	**Our results**	**Standard results**
Diffraction Angles	37.96,43.93,58.65,62.10,73.98	38.43,44.37,59.17,64.52,73.33
Grain Size	21.79	20
Miller Indices	111,200,211,220,310	111,200,220,311
Structure	Cubic	Cubic
Lattice Constant	4.15	4.21
Strain	Large	Small
Density	3.74	3.58

This represents a very small difference between our results and the standard results and this difference is only because we avoid the basic necessary conditions of preparation such as, vacuum furnace, required pressure and automatic control system.

3.2. UV-visible Analysis

UV-visible at Different Annealing Temperatures

Absorbance spectra for Magnesium oxide at different annealing temperatures are shown as:



This spectra represents that with the increase in annealing temperature absorbance peaks are shifted toward the lower wavelength and also represents that the peaks are obtained in the visible region at 774,764,756 and 750 nm respectively.

Annealing Temperature	Percentage Yield	Absorbance Peaks
25℃	40%	774nm
70℃	44%	764nm
210℃	57%	756nm
450℃	83%	750nm

Discussion with UV-visible Result

Using UV-visible we found.

- Grain size
- Absorption

- Reflectance
- Transmission
- Band Gap
- Dielectric
- Conduction

❖ ***Grain size***

Grain size of magnesium oxide (MgO) we can calculate by using effective mass model

$$\mathbf{E}=\boldsymbol{E_g}+\frac{\hbar^2\pi^2}{2r^2}\left(\frac{1}{\mathbf{m_e}}+\frac{1}{\mathbf{m_h}}\right)\text{..... (1)}$$

Where

E_g=Bulk energy gap (7.8eV for MgO)

r=radius of particle

m_e=Effective mass of electron ($0.24m_0$)

m_h=Effective mass of holes ($0.45m_0$)

$$\boldsymbol{r^2}=\frac{\hbar^2\pi^2}{2(E-E_g)}\left(\frac{1}{\mathbf{m_e}}+\frac{1}{\mathbf{m_h}}\right)\text{..... (2)}$$

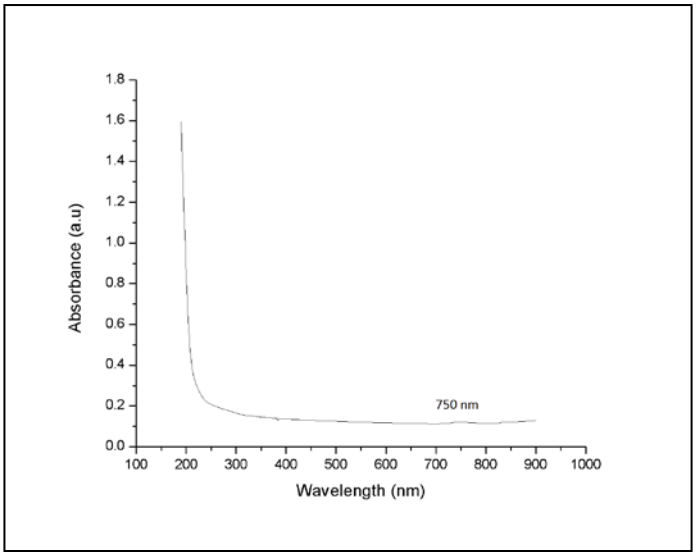


Table 18: Grain Size

Energy(E)	Bulk Energy(E_g)	Grain Size(nm)
1.653eV	7.8eV	~1nm

Range of Nanoparticles for MgO using UV-visible is about 1nm. UV-visible gives us the range of small size Nanoparticles.

❖ ***Absorbance***

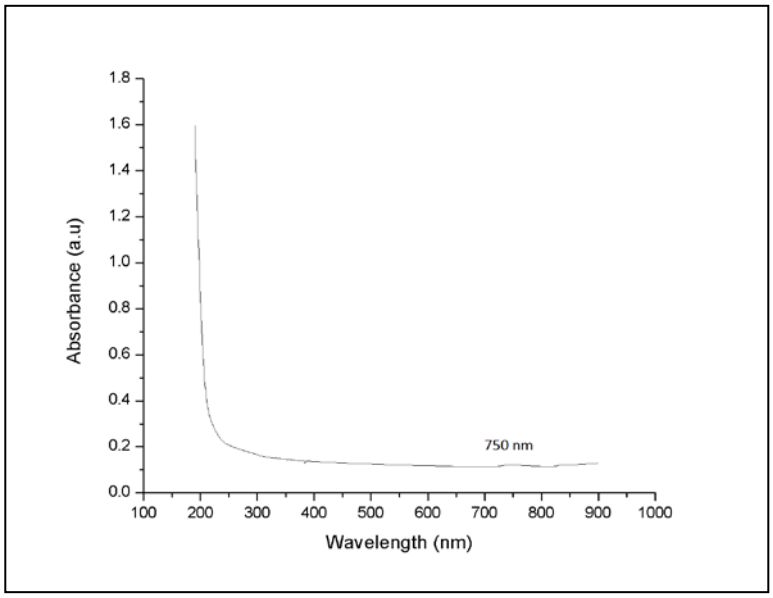
Absorbance is the percentage of incident light absorbed by the sample or a part of incident beam which is neither transmitted not reflected. Absorbance is inversely proportional to the transmission as.

$$\alpha \propto \frac{1}{T}$$

$$A=\alpha=\frac{log\left(\frac{1}{T}\right)}{d}$$

Where

A=Absorbance, T=Transmission, d=1



In UV-spectra absorption for MgO is observed at **750nm.**

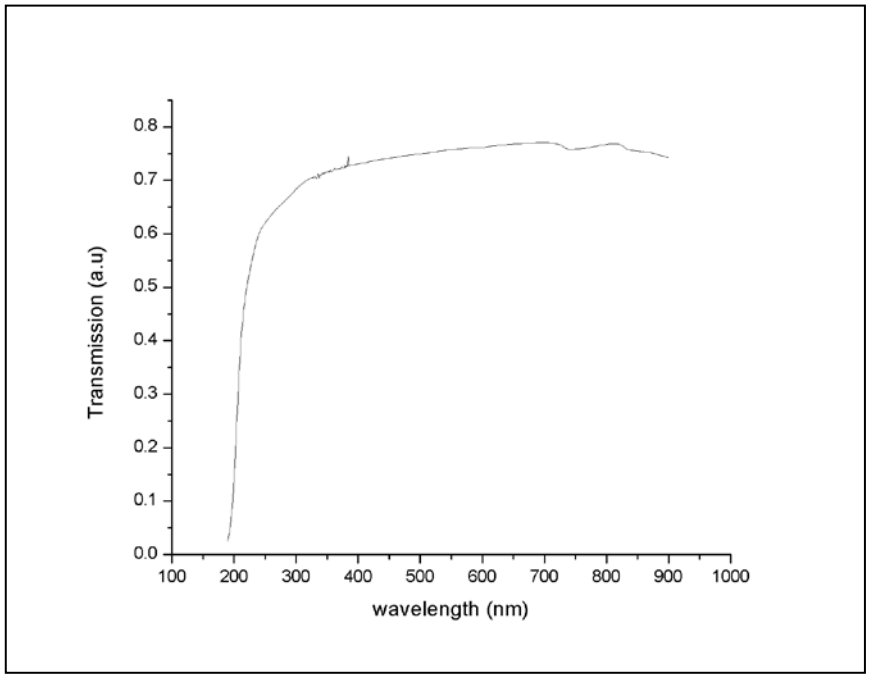
❖ ***Transmission***

Transmission is the percentage of incident light passes through the sample. Transmission is the reciprocal of the

absorbance and we can calculate as.

A=log ($\frac{1}{T}$)

T=e^{-A}=10^{-A}



In UV-spectra transmission for MgO is observed at **750nm.**

❖ ***Reflectance***

Reflectance is the percentage of incident light reflected back at different angles when light is incident on the sample and we can calculate as.

R+A+T≤1

R=1-T-A

❖ ***Band Gap***

The direct band gap energy for magnesium oxide is obtained by fitting a relation between $(\alpha h\upsilon)^{\frac{1}{n}}$ and A $(h\upsilon - E_g)$.

By using Tauc's relation we can find"E_g".

$(\alpha h\upsilon)^{\frac{1}{n}}$**=A (hυ − E_g)……. (1)**

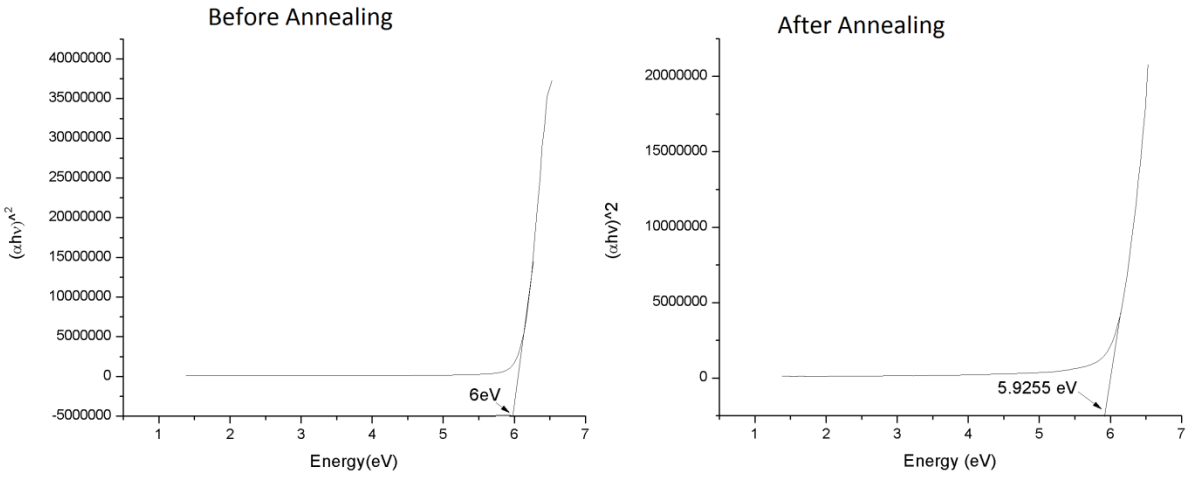
Now plot a relation between $(\alpha h\upsilon)^{\frac{1}{n}}$ and $h\upsilon$,from this relation we can find "E_g" by extrapolating a slope to make y-axis equals to zero.

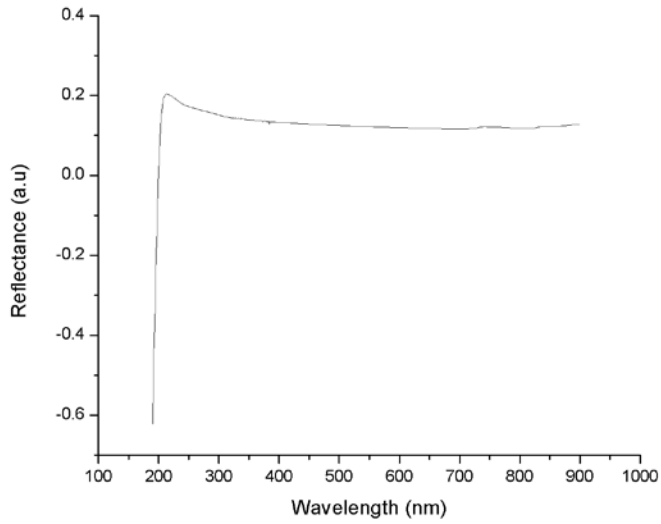
Equation (1) can be written as

0=A (hυ − E_g)

$\boldsymbol{E_g}$**=hυ**

The band gap energy for magnesium oxide after annealing at 450℃ is found to be **5.92eV** and the band gap energy before annealing is about **6eV**. It means band gap energy decreases with the increase in temperature for MgO.





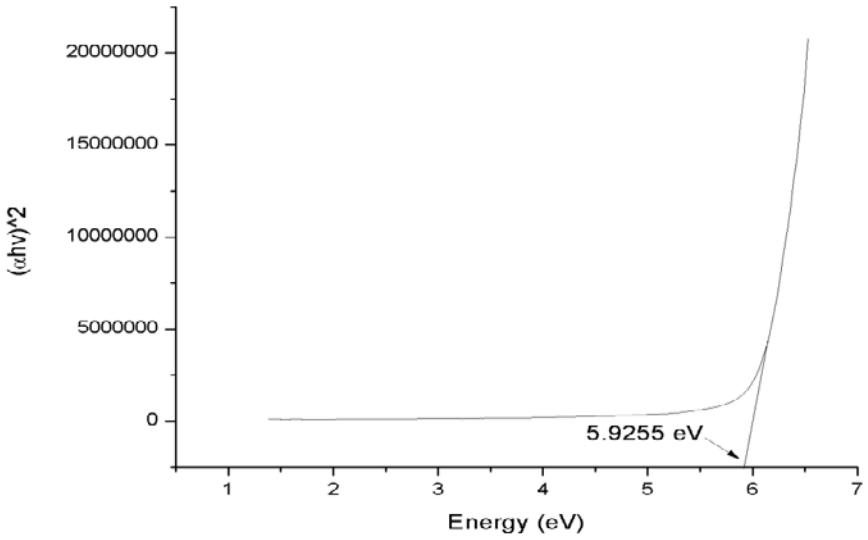


Table 19: Comparison Before and After Annealing

Before Annealing		After Annealing	
Wavelength	Band gap energy	Wavelength	Band gap energy
774nm	6 eV	750nm	5.92 eV

❖ ***Conduction***

The observed band gap decreases with increase in annealing temperature for **MgO**, decrease in band gap represents **increases in conduction**. It means MgO annealed at **450 C** is good conductor than **MgO** before annealing.

Discussion with SEM Result

The term SEM is derived from scanning electron microscope which we used to analyze the structure, morphology and grain size of magnesium oxide Nanoparticles. The instrument was accelerated at about 20KV and scanned the sample at a distance of 15mm.The sample was dispersed in isopropyl alcohol and scanned with different magnifications at 1,500x, 5,000x and 10,000x.SEM images as given below tell us the grain size for pure magnesium oxide that is in the range from 20-50 nm. SEM images are given below

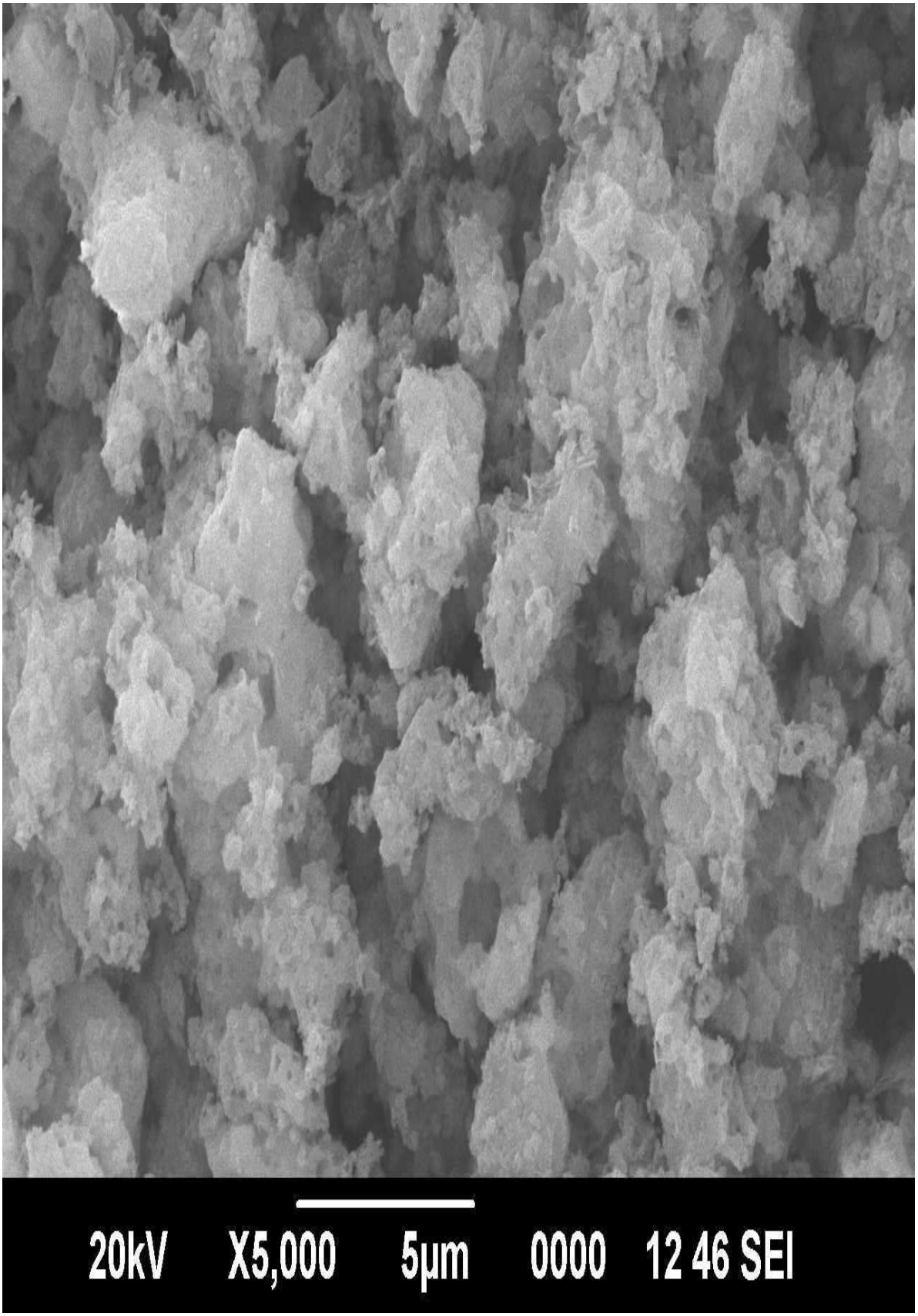


Figure 7.a: (SEM with magnification X1,500).

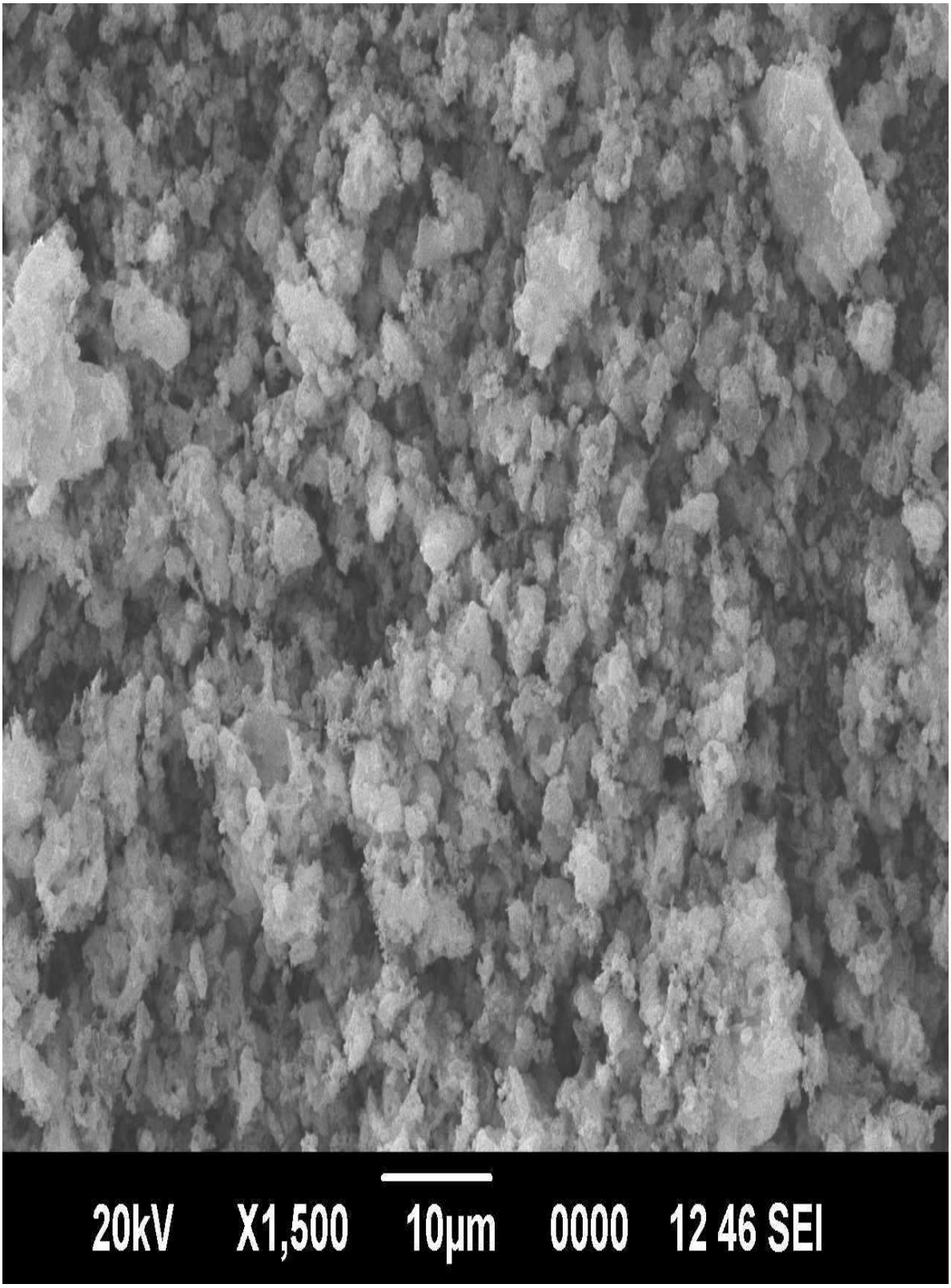


Figure 7.b: (SEM with magnification X 5,000)

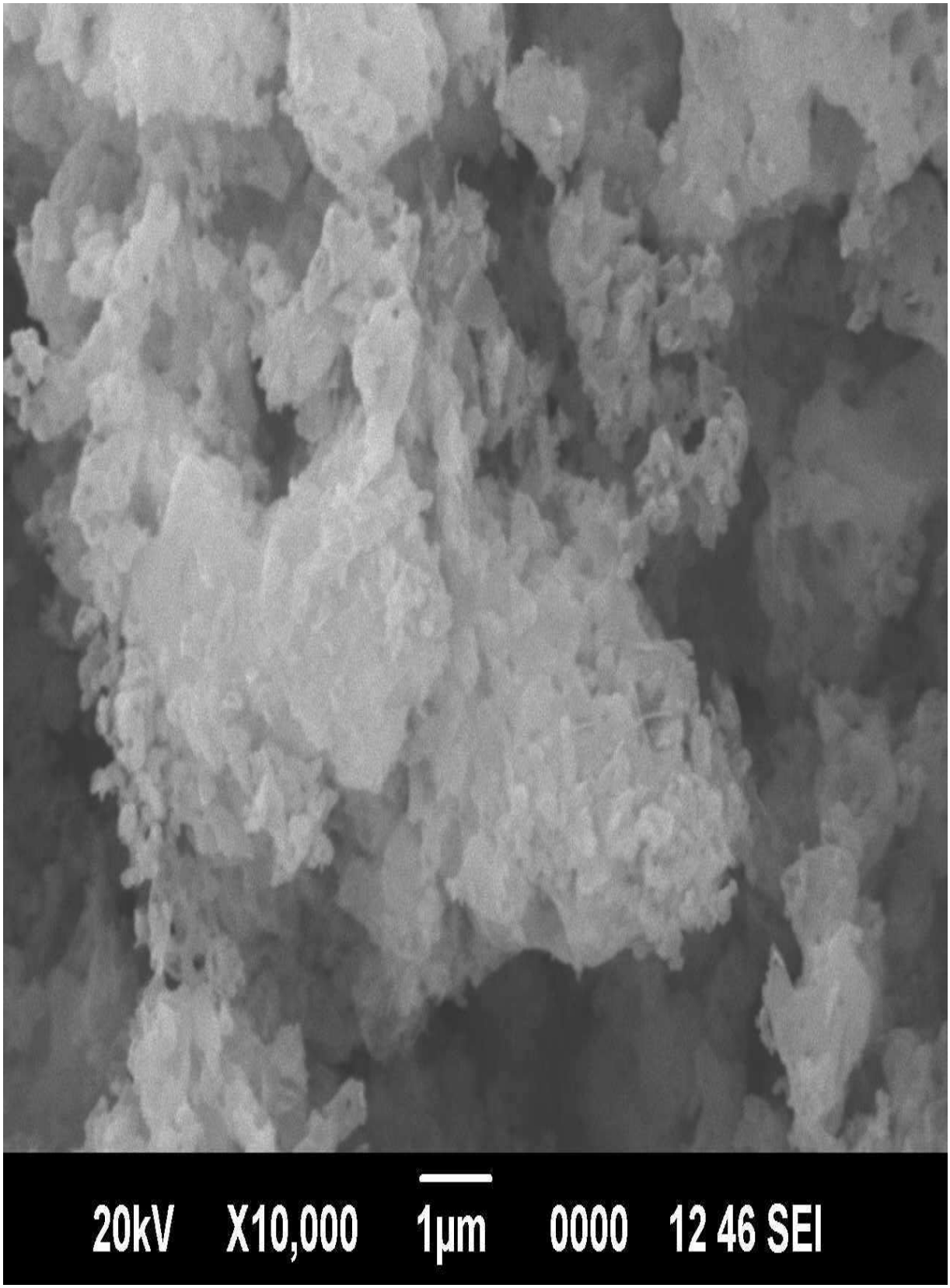


Figure 7.c: (SEM with magnification X 10,000)

4. Conclusion

Magnesium oxide (MgO) Nanoparticles were synthesized by using the CHM method in this method we had introduced annealing process for the preparation of oxides of ⅡA group elements, just because we had no vacuum furnace. By using vacuum furnace with required temperature and pressure we can prepare magnesium oxide (MgO) and other oxides of ⅡA group such as BeO, CaO, SrO, BaO without annealing process but CHM without Vacuum furnace is best because this way is cheap and easy to handle as compare to CHM with vacuum furnace and as compare to other methods. Synthesized Nanoparticles were characterized by XRD, UV-vis spectroscopy and SEM, by using results of XRD it was observed that due to annealing, diffraction angles slightly shifted from their original and standard position, Shifting in diffraction angles is because of the presence of stabilizing aluminum impurities. It was also observed that there is slight change in grain size, decrease in lattice constant, shifting of Bragg planes, increase in strain, increase in dislocation density and increase in density.

By using CHM method we obtained the ideal size of MgO Nanoparticles with size of 21nm by using other methods of synthesis we mostly obtained the MgO Nanoparticles with size > 20nm,from this method we achieved the best Nanoparticles of MgO which can be used as best Antibacterial agent.

The bactericidal efficacy of nano-MgO increased slowly with decreasing particle size. Below ~ 45 nm however, the bactericidal efficacy showed a much stronger dependence on particle size. Makhluf *et al*. (2005) demonstrated that small MgO nanoparticles had an efficient antibacterial activity towards *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*) [7].

In UV-vis spectroscopy we observed general range of grain sizes for MgO and it was also observed, absorption peak is at 750nm also the reflectance and transmission peaks are at 750nm,and band gap energy is 5.92 eV, the decrease in band gap energy for MgO is observed with the increase in annealing temperature, it means conduction increases.

In SEM we observed the accurate size of grain for MgO that is about 20nm

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