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Synthesis and Structural Characterization of MgO Nanoparticles

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ABSTRACT:Magnesium oxide (MgO) nanoparticles are synthesized by Sol - Gel method using $Mg(NO_3)_2.6H_2O$ as a precursor and different molar concentrations of NaOH as a stabilizing agent. X-ray diffraction pattern (XRD) reveals single phase fcc structure with a lattice constant 4.21Å . Scanning electron microscopy (SEM) showed the aggregated irregular and tiny crystals of as prepared MgO nanoparticles. By using Williamson-Hall equation , the particle sizes vary within the range of 11.27 -14.44 nm.

KEYWORDS:Nanoparticles, Sol-Gel Route, Magnesium oxide , XRD , SEM

I. INTRODUCTION

Nano size materials have different properties compared with bulk materials. Nanoparticles are particles between 1 and 100 nanometers in size and it are having attract a great attention in recent years because of their unique electronic, physical, magnetic, chemical and optical properties compare with bulk materials[1, 2]. Magnesium oxide is an inorganic compound having thermal stability, high surface reactivity, very good heat resistance, high chemical and alkali resistance[3, 4].MgO oxide has a wide range of applications such as superconducting products, toxic waste remediation, ferroelectric material, catalysis, catalysts support, paint, adsorbent or pH regulator for wastewater treatment and high activity against bacteria [5-8]. Different methods are available to prepare MgO-NPs such as vapor-liquid-solid (VLS), solution combustion, chemical vapor deposition (CVD), wet co-precipitation, plasma enhanced, quick precipitation route, chemical vapor deposition (PECVD), sol-gel, pulsed laser deposition (PLD), hydrothermal route, green synthesis, laser ablation, solvothermal, molecular beam epitaxy (MBE), microwave assisted sol-gel and sputtering method [9-14]. Oxygen is VIA group element with atomic number 8 and Magnesium is IIA group element with atomic number 12. The compound MgO is having melting and boiling points as 2852ºC and 3600ºC.

In the present study, we have synthesized MgO nanoparticles by sol-gel method and to characterize MgO-NPs by XRD, SEM and Rietveld refinement techniques.

II. EXPERIMENTAL

A.Synthesis

The synthesis of MgO nanoparticles is divided into various steps, such as mixing, stirring, filtering, drying and calcination. Finally by the calcinating the powder at 600 ^oC for 3 h, then MgO is obtained in the nanoparticles .

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Initially $Mg(NO_3)_2.6H_2O$ of wt. 5.21 g (0.1 M) and dissolved in 200ml of distilled water. The 0.8 g (0.1 M) of NaOH in 200ml distilled water. Then 200ml of NaOH solution is added in solution of $Mg(NO_3)_2.6H_2O$ drop-wise by using glass rod. After that, solution kept under magnetic stirring for 3 h after stirring the solution was kept on table at rest for 3 h so that, the precipitation is formed at the bottom of beaker. This precipitation was filtered and washed several times by using distilled water so as to get the final products. The final product was kept in oven at 100 °C for 6 h for drying product and removing the moisture. This dried powder is then crush and make it very fine powder by using mortal pestle. Finally the fine powder of MgO is calcinated at 600 ^oC for 4 h for the removal of impurities present in the powder , according to the following equation.

Mg (NO3)2 .6H2O + 2 NaOH → Mg (OH) 2 + 2NaNO3

Mg (OH) 2 $\xrightarrow{\Delta}$ MgO +H2O

So that we will get synthesized MgO possessed high crystallinity with the particle size in nanosized range. After that the same steps be returned to prepare other samples with a concentration of NaOH 0.15M,0.2M,0.25M ,they named as S1,S2,S3,S4 respectively .

B.Characterization

The crystalline structure of the nanoparticles was determined by X-ray diffraction (Shimadzu XRD-6000) with Cu Kα1 radiation. The nanoparticles morphology was observed using a (VEGA\\Easy Probe) scanning electron microscope. Rietveld analysis was carried out to calculate unit cell parameters.

III. RESULTAND DISCSSION

A-XRD measurement

The XRD pattern of all the samples, MgO-NPs as prepared are shown in figure (1). They were compared with JCPDS file No. (45-0946) and no any crystalline impurities were detected. The observed diffraction peaks correspond to the cubic crystalline MgO with space groupe (F m $\bar{3}$ m). The results of diffraction angle (2Θ), full width at half maximum (FWHM) β, (d) spacing between crystal planes for three strongest peaks are shown in table (1).

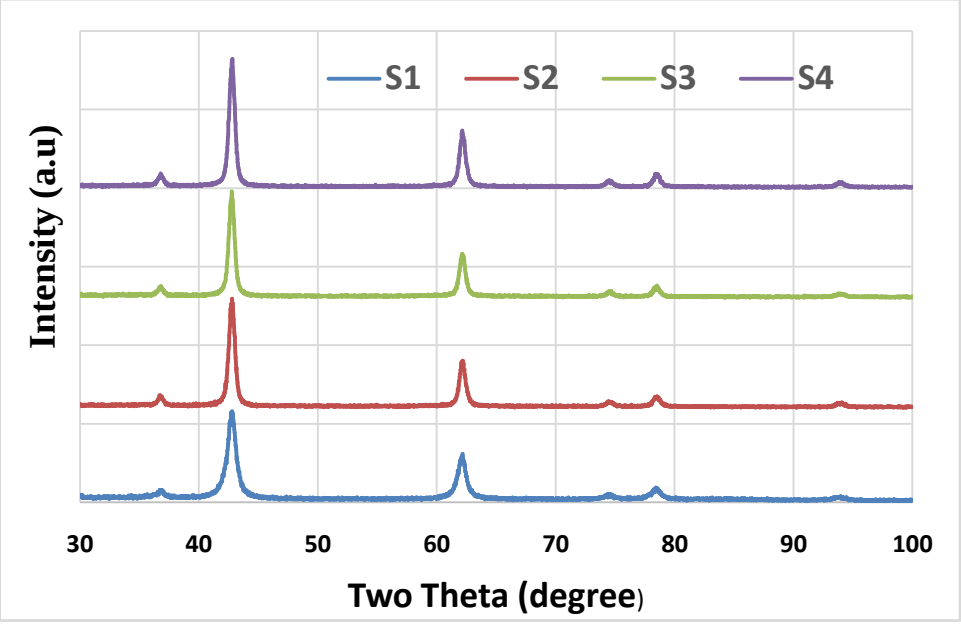

Fig. 1. XRD pattern of MgO-NPs

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Vol. 3, Issue 7 , July 2016

Table 1. XRD data of MgO-NPs

Sample	2Θ(˚)	d(Å)	β(˚)	I/I_1
S1	42.7581	2.11310	0.84380	100
	62.0786	1.49391	0.81340	49
	78.3790	1.21905	0.85340	12
S2	42.7736	2.11237	0.60550	100
	62.1341	1.49271	0.57900	44
	78.4365	1.21830	0.63500	9
S3	42.7551	2.11324	0.57140	100
	62.1266	1.49287	0.56900	41
	78.4290	1.21839	0.62000	9
S4	42.7790	2.11211	0.59130	100
	62.1390	1.49261	0.57610	44
	78.4415	1.21823	0.61500	10

B. Crystallite Size Calculation

The crystallite size and lattice strain induced in powders arising from defects like distortion, twinning, imperfection and dislocation was determined by Williamson-Hall equation [15]:

$$\beta_{hkl}\cos\Theta = (K\lambda / D) + (4\varepsilon \sin\Theta) \quad \ldots\ldots(1)$$

where D: crystallite size, K: constant, λ: wavelength of Cu kα radiation.
W-H plot is shown in figure (2).

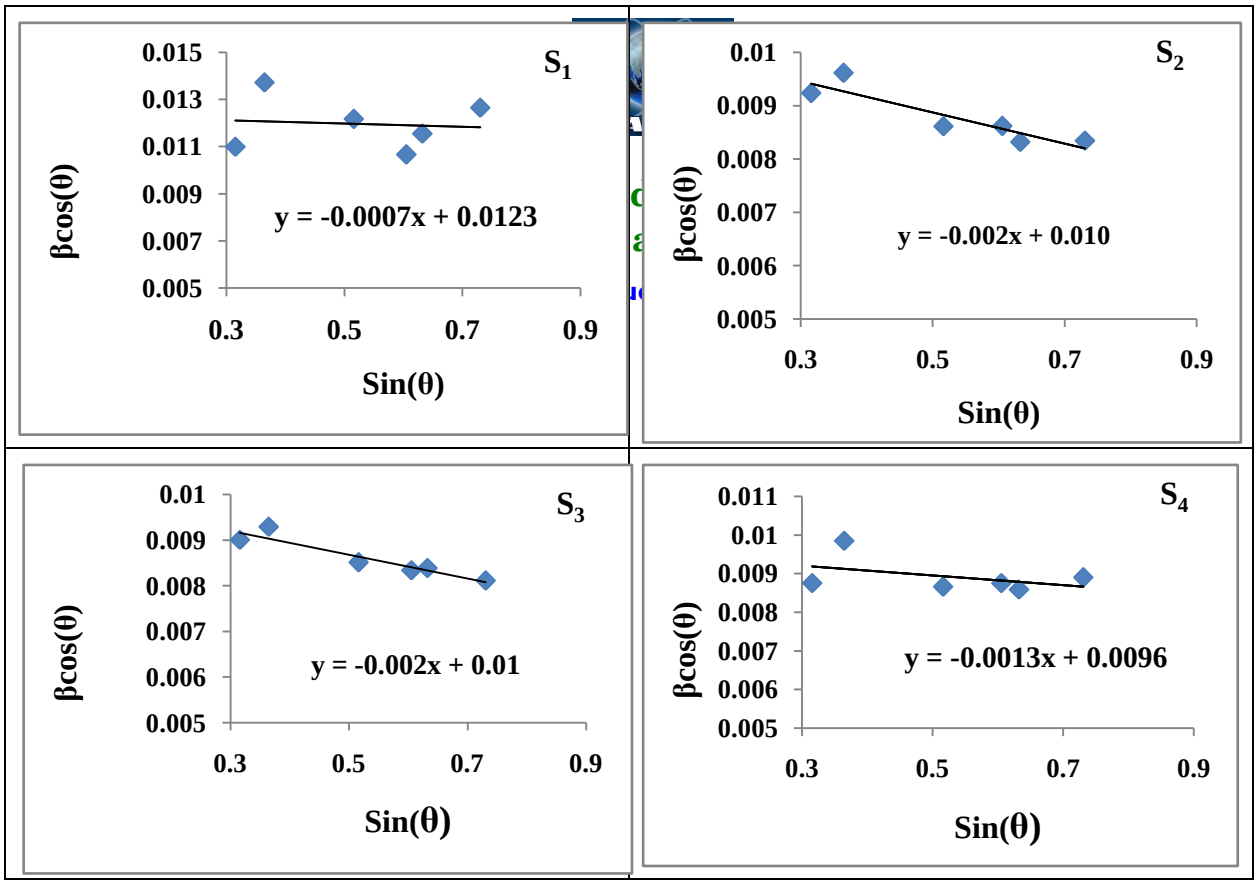

Fig. 2. Williamson Hall plot of MgO-NPs

It is plotted with sinΘ on the x-axis and β_{hkl}cosΘ on the y-axis (β_{hkl} in radian).Table (2) shows strain and crystallite size according to W-H for MgO nanoparticles.

Table 2. Strain and crystallite size according to W- Hof MgO-NPs

Sample	D (W-H)nm	ε (W-H)*10^{-4}
S_1	11.27	-1.75
S_2	13.46	-7.25
S_3	13.86	-6.5
S_4	14.44	-3.25

From table (2) it shows that the particles size increase with increase concentration of (NaOH) because of decreases in the density of nucleation centers lead to a smaller number of centers start to grow, resulting in large crystallites [1].

C. RietveldRefinment

Rietveld refinement is a technique used to solve a structure crystalline materials from the powder diffraction data; discovered by Hugo Rietveld. The results powder diffraction from neutron and X-ray are characterized by reflections (peaks in intensity) at certain positions. The position, height and width for peaks can be used to determine the material's structure. The Rietveld method uses a least squares to reduce the difference between the calculated and observed patterns[16–18].Rietveld refinements of MgO-NPsfor S1, S2, S3 and S4 are shown in figure (3) and the results of indexing and refinement are shown in table (3).

ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 3, Issue 7 , July 2016

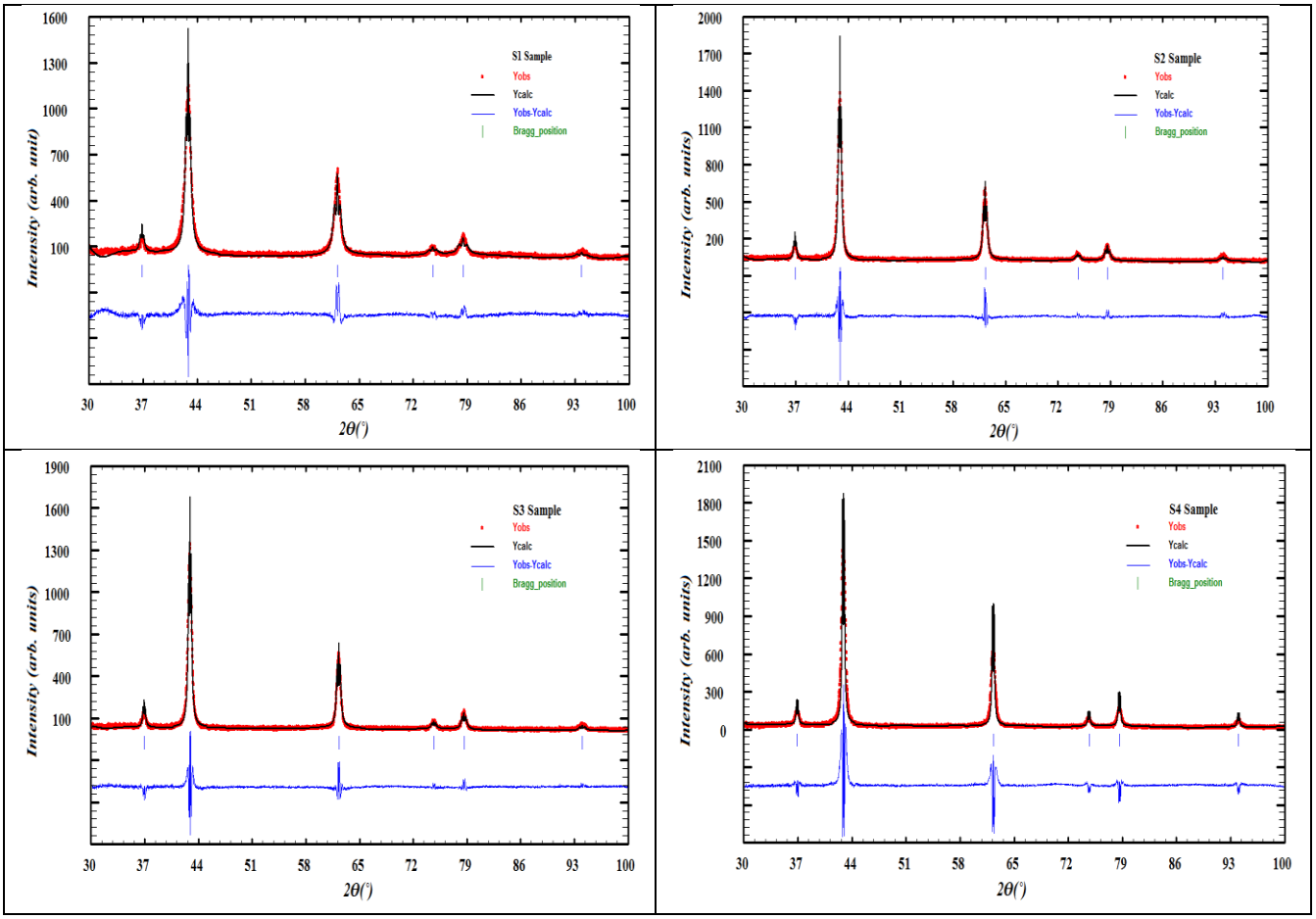

Fig. 3. Rietveld refinements of MgO-NPs for S1, S2, S3 and S4 samples.

Table 3. The results of indexing and refinement for MgO-NPs.

Sample	Unit cell parameters (Å)	Crystal System	Space group	χ^2
S1	a	fcc	F m $\bar{3}$ m	2.80
	4.2184			
S2	4.2115	fcc	F m $\bar{3}$ m	2.83
S3	4.2116	fcc	F m $\bar{3}$ m	2.74
S4	4.2170	fcc	F m $\bar{3}$ m	5.57

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Vol. 3, Issue 7 , July 2016

D. SEM images analysis

The SEM micrographs for MgO-NPsS1, S2, S3 and S4 are shown in figure (4).

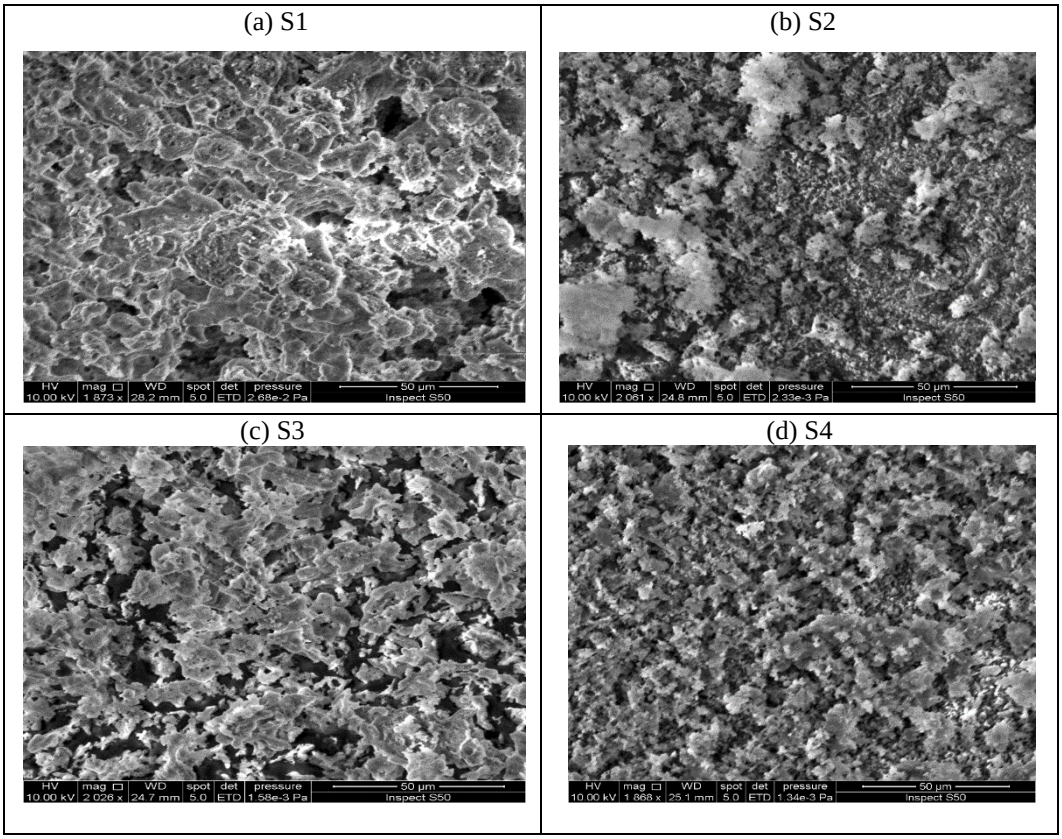

Fig. 4. SEM images of MgO-NPs

The SEM micrographs (Fig. 4a) show the particle morphology of MgO consist of aggregated irregular and tiny crystals. Fig. (4b) shows with increasing the concentration of NaOH the more agglomeration occurs among particles. Fig. (4c) MgOnanomaterials are seemingly porous and highly agglomerated with fine particles. Due to the porous nature of MgO nanoparticles may use gas sensing application.Fig. (4d) show that the porosity less than the rest of the samples, this means that the particle size of this particle is greater than the others sample. This is agree with W-H results.

VI. CONCLUSION

Magnesium oxide nanoparticles (MgO-NPs) with a cubic structure have been prepared successfully by sol gel method. The average particles size are estimated by W-H formula of MgO-NPs for all samples in nanoscale. The average crystallite size increase with increase concentration of (NaOH). The results of XRD and Rietveld analysis have a good agreement. The SEM micrographs of MgO-NPs for S1, S2,S3 and S4 show the porosity decrease with increase in NaOH concentration and with a high degree of agglomeration among fine particles.

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International Journal of Advanced Research in Science, Engineering and Technology

Vol. 3, Issue 7 , July 2016

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