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MORPHOLOGICAL VARIATION WITH MGO DOPANT CONCENTRATION IN $MGAL_2O_{4-\Delta}$ NANOSTRUCTURED MATERIAL (NSM)

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ABSTRACT
The effect of concentration on structural and morphological has been examined for the magnesium aluminate ($MgAl_2O_4$) nanostructured materials. In the current study, nanosized $MgOAl_2O_3$ samples were prepared by microwave assisted co-precipitation method. The particle size is evaluated using an X-RD method with varying concentration of magnesium in magnesium aluminate nanoparticles. The crystallite size of the nanoparticles is calculated using the Debye- Scherrer' equation and observed that particle shows inverse characteristics with rise of dopant concentration. Moreover , the morphology and purity were examined by using HRTEM and IR spectroscopic techniques respectively. The IR peaks at around 3000 cm^{-1},1650cm^{-1} are due to OH vibration and peak at 528.6 cm^{-1} confirmed Alumina (Al-O) vibration whereas, peak occurred at 810 cm^{-1} are due to O-Mg-O vibration and are proving that Mg^{++} ion is incorporated in Al_2O_3 structure. The HRTEM images shows that particle are truncated spherical shape and agglomerated in nature.
Key words: Magnesium aluminate Spinel (MAS), Debye Scherrer equation, XRD, FTIR, HRTEM etc.

INTRODUCTION
Nanotechnology is a rapidly growing branch of today science that deals with materials at the nanoscale, i.e. from 1-100 nm. Due to their nanosize, nanomaterials exhibit unique properties compared to bulk materials of the same element or compound. At nanoscale, the intrinsic properties of materials can be selectively altered in successive steps of fabrication, with numerous properties dependent on the material's grain size [1]. The progress of human civilization is highly dependent on the development of noval technologies in the field of material science to improve social life. Among the various materials currently in existence, eg ceramics, concrete have played an important role in the advancement of human civilisation [2]. Normally ceramics and advanced ceramics are the main types of ceramic materials. Traditional ceramics are made from natural materials such as quartz and feldspar, while advanced ceramics are made from synthetic powders such as silicon nitride, aluminum oxide such as inorganic materials. At present, there are many types of ceramic materials but spinel structure materials are attracting a lotsof attention due to their remarkable applications in the scientific and technological fields. [3]. Spinal aluminates play an important role in solid state science due to their excellent electrical and optical properties

and also show remarkable chemical and magnetic properties. The crystallite structure of spinel materials includes the basic chemical composition AB_2O_4. where A and B are divalent and trivalent element respectively.[4] There are mainly 200 types of compounds with a spinel structure and are usually requirement of daily life of living world and industrial field. Magnesium aluminate is a promising material with an excellent combination of physical and exceptional chemical and synthetic properties and can be used as a refractory oxide and as a displacement agent of harmful radiation due to its high resistance to radiation damage. Among all the spinel structural materials, $MgAl_2O_4$ has received special attention due to its environmental friendly nature. [5] In the crystal structure, magnesium ions occupy tetrahedral sites and aluminium ions occupy octahedral sites. The crystal structure has up to 64 octahedral sites and 32 tetrahedral sites. By stretching in three directions and doubling each fcc oxygen sublattice, a regular spinel unit cell is formed [6]. As the composition changes, the arrangement of the metal ions at the octahedral and tetrahedral sites changes. The high melting point (M.P) and resistance to acid and alkali elements and l excellent properties of MAS nanomaterials have many industrial applications [7]. In this chapter, nanostructured $MgAl_2O_4$ spinel materials are analysed for their structural and optical properties using various characterizations techniques. Structural analysis was performed using X-ray diffraction, Fourier transform infra-red spectroscopy [8].

Experimental Details

Co-precipitation method followed by microwave was used to synthesize the desired samples. In this method, magnesium aluminate nanomaterials were synthesized by dissolving appropriate amounts of aluminium nitrate and magnesium chloride in 100 mL of deionized water. The resulting precursor was completely refluxed by using a stirrer. The addition of the ammonium solution to the resulting compound mixture was dropwise to facilitate the precipitation reaction. The precipitates were filtered using good quality filter paper and washed with distilled dihydrogen oxide to remove excess ammonia and change the nature of the solution from acidic to basic. Excess water impurities present in the precipitates were removed by placing it on a hotplate at 100°C for 4-5 hours. The prepared samples were crushed by using an agate-motor & pestle. Thereafter, calcined at 800ºC for fixed duration of 2 hrs and to investigate the effect of concentration changes on its optical and structural properties of the calcined samples.

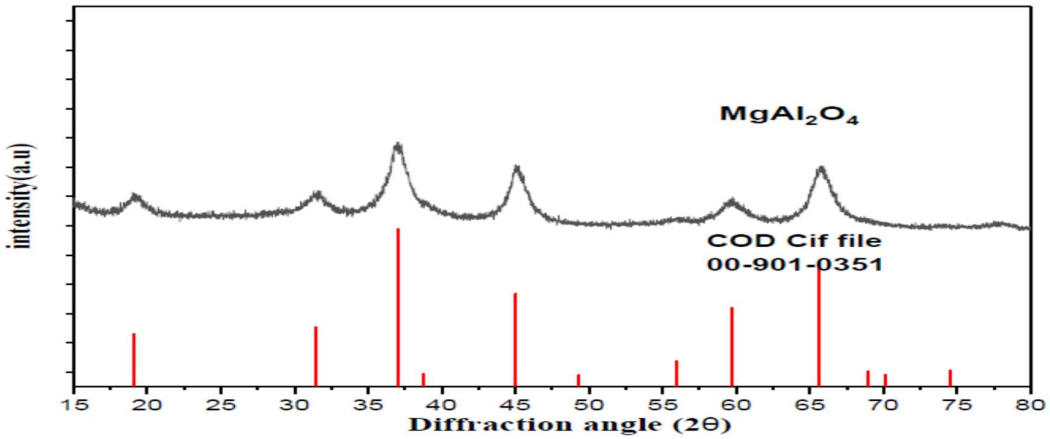
Results and Discussion

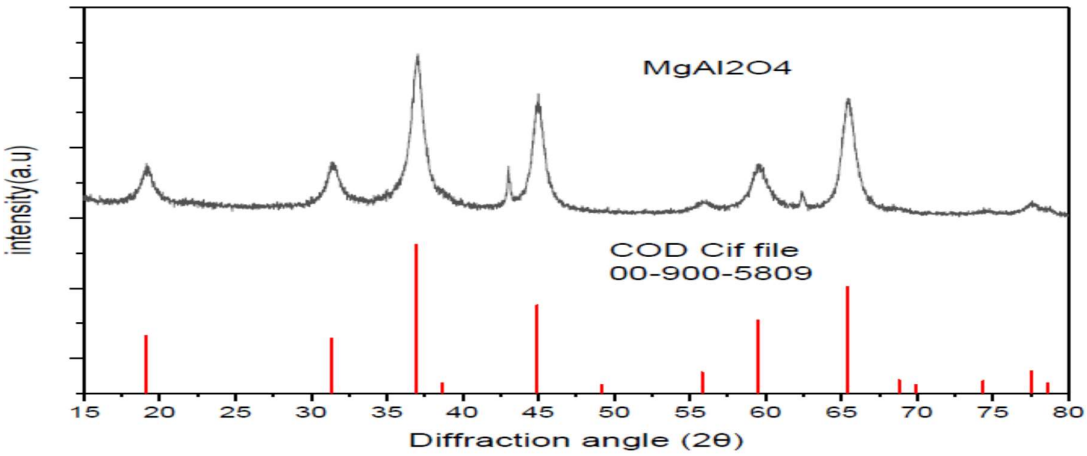
Structural characterization:

X-ray diffraction analysis was performed using copper Kα radiation with a wavelength of 0.154 nm. Structural information is obtained by exposing the powder form of the sample with an X-ray beam and then examining the diffracted beam to determine the crystallite size of the nanoparticles (NP's). The respective positions of the atoms on the particular sites were obtained by using the diffraction pattern of the as synthesized nano- samples. X-ray line profiles of powder samples are basic tools for its crystallographic study. This is because the peak width (FWHM) and its intensity provide many useful parameters for structural analysis such as Phase identification, defects in crystal structure, unit cell size, lattice constant etc. Diffraction patterns of MgO doped Al_2O_3 nanostructured materials with different doping element compositions are shown in figure 1. The study reveals that FCC

crystalline structure with lattice constant a=b=c 8.06 were occurred for Al_2O_3-MgO (10%) sample calcined at 600ºC for 2 hours whereas, small changes were recorded for Al_2O_3-MgO (10%) calcined at 600ºC for 2 hours i.e a=b=c= 8.045. It may be caused by strain in lattice with increase of dopant concentration.

(a) **Figure1:XRD pattern of Al_2O_3-MgO (10%) samples calcined at 600ºC for 2 hours.**





(b)

(b)Figure2: XRD pattern of Al_2O_3-MgO (20%) samples calcined at 600ºC for 2 hours

The calculated crystallite size of the major (high) peak sample is calculated by using Debye Scherrer equation: i.e

$$D=\frac{K.\lambda}{\beta Cos\theta}$$

Where D is crystalline size in nm, k is Scherrer shape factor and for the spherical shape the value is 0.89, λ is the wavelength of k_α copper radiation with wavelength of 0.15 nm, β is the full width at half maximum intensity (FWHM)for main (major) peaks in radians and θ(theta) is the diffraction angle [10]. The particle sizes of the strongest peaks are 16.92 nm for 10% dopant concentration and 10.68 nm for 20 % dopant concentration calcined at 600ºC for 2 hours. The decrease of crystallite size with rise of dopant concentration indicated that the ionic radii of Mg^+ ions is less as compared to Al^{3+} ions.

Table1. Various parametric study of MgO doped Al_2O_3 (10%) calcined at 600ºC for 2 hours

Peak position(2 θ)	FWHM(β)	hkl plane	d-spacing
19.2	0.010777135	111	4.623
31.4	0.011668799	220	2.845
37.1	0.009030806	311	2.421
44.9	0.009626764	331	2.015
59.6	0.015176911	511	1.554
65.5	0.012558917	440	1.425

Table2. Various parametric study of MgO doped Al_2O_3 (20%) calcined at 600ºC for 2 hours

Peak position(2 θ)	FWHM(β)	hkl plane	d-spacing
19.2	0.010776134	111	4.641
31.3	0.011667798	220	2.856
37.0	0.009030807	311	2.425
38.7	0.005541146	222	2.323
45.1	0.009625765	400	2.012
59.6	0.015157912	511	1.551
65.6	0.012557918	440	1.421

FTIR Spectroscopy analysis

The IR spectroscopic tools are employed to find the functional groups and chemical bonds present in various samples, and the results of X-ray diffraction data are also confirmed [11]. The main transmission peaks for the 10% dopant concentration observed around wavenumbers of about 3430, 1482, 707 and 544 cm^{-1} are shown in Figure 4. and the main peaks at 3464, 1638, 1425, 709 and 528.6 cm^{-1} for 20% MgO doped Al_2O_3 composition are shown in Figure 4. Incomplete decomposition of nitrate explained by the band present around 1425 cm^{-1}. Vibration modes of water, i.e. Oxygen-hydrogen bonds are confirmed by the broad peak observed around 3460 cm^{-1}. There were broad peak observed at IR spectrum at position 868cm^{-1}, 709cm^{-1} and 528 cm^{-1} and it might be due to O-Al-O and O-Mg-O vibrations of MgO doped Al_2O_3.

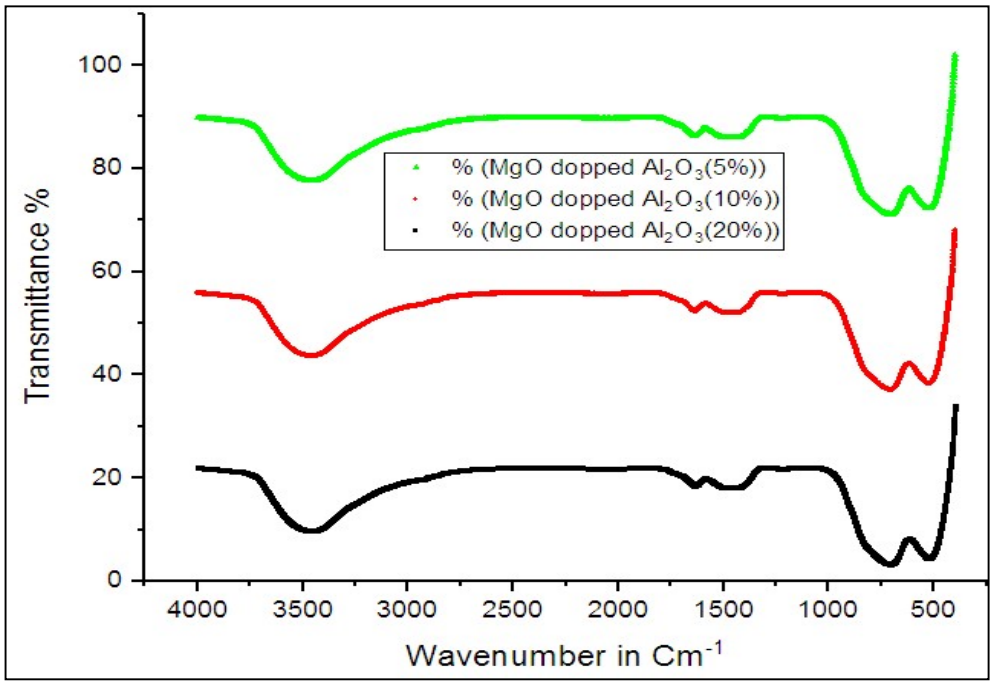


Figure 4: The IR spectra of samples calcined at 600ºC for 2 hours (a) MgO doped Al_2O_3 (5%) (b) MgO doped Al_2O_3 (10%) (c) MgO doped Al_2O_3 (20%)

High resolution Transmission electron microscopy (HRTEM) analysis technique:

The analysis of high resolution Transmission electrons analysis techniques shows that the surface (behavior) investigation of the magnesium doped Al_2O_3 Nano stuff in **figure5** indicate that size of nano-particles is in nanometer range (1-100nm) and reflectivity is varying with dopant concentration of magnesium into Al_2O_3 Nano particles. The HRTEM images shows that the particles are more less truncated spherical in shape and agglomerated in nature.

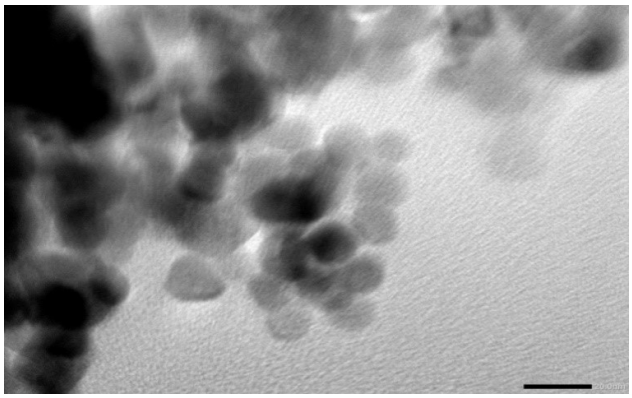


Figure 5 : HRTEM images of Mg 10% doped Al_2O_3 calcined at 600ºC for 2hrs

CONCLUSION

The various concentration of MgO doped Al_2O_3 NSM were synthesized by microwave modified co-precipitation techniques and samples were calcined for 600ºC for fixed duration of 2 hours. The XRD study reveals that crystallite size of samples calcined at 600ºC for 2 hours

were decreased with rise of dopant concentration from 16.38 nm (10%) and to 10.68 nm (20%). The IR spectroscopy concluded that the IR peak position exhibited at 868cm^{-1}, 709cm^{-1} and 528cm^{-1} were attributed to O-Mg-O & O-Al-O vibration of these groups present in $MgAl_2O_4$ samples respectively. The morphology of samples were examined by HRTEM and found truncated spherical in shapes.

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