

Preparation and Characterization of Magnesium Aluminate Nanoparticles by Hydrothermal Method

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Abstract

Magnesium aluminate is a compound with a negatively charged alumina ion and metallic oxide with various industrial applications such as water treatment and ceramics manufacturing. In this research, magnesium aluminate nanoparticles were synthesized by hydrothermal-assisted sol-gel method. Magnesium aluminate gel was prepared by using aluminium nitrate, magnesium chloride and urea. The resulted magnesium aluminate gel was calcinated at different temperatures for 4 hours and then were characterized by using modern techniques (XRD, FT IR and SEM). XRD data of the MgAl_2O_4 nanoparticles showed characteristic peaks related to miller indices of 111, 220, 311, 400, 511, 440 and 531, these peaks are well matched with standard library data of JCPDS (77-0438>spinel- MgAl_2O_4). By using Scherrer equation, crystalline sizes of the MgAl_2O_4 nanoparticles obtained at calcination temperature of 800, 1000 and 1150°C were 10.15, 25.77 and 28.02 nm, respectively. FTIR spectra of MgAl_2O_4 nanoparticles were recorded and studied in the wave number range 400-4000 cm^{-1} . The band over the range of 1000-400 cm^{-1} corresponds to metal-oxygen bonds (Al-O and Mg-O). SEM microphotograph of MgAl_2O_4 nanoparticles showed as flake-like shapes.

Keywords: MgAl_2O_4 , hydrothermal, XRD, FT IR, SEM, Scherrer

Aim

To study simple, reliable and inexpensive production method for magnesium aluminate nanoparticles

Introduction

Magnesium aluminate spinel is a member of a group of oxides that have the same crystal structure, which is named the spinel structure (Schmidtmeier *et al.*, 2009). Magnesium aluminate spinel (MgAl_2O_4) is one of the best known and it is widely used in polycrystalline materials. It possesses a good combination of features such as high melting point, good mechanical strength, low dielectric constant and high resistance against both alkalis as well as acids (Chandradass and Kim, 2010).

Nomenclature, formula and characteristics

Accepted names	:	Magnesium Aluminate, Magnesium Aluminium Oxide
Classification	:	Ceramic, metal oxide
Apperance	:	White, (red, blue, green, yellow, brown or black)
Odour	:	Odourless
Empirical formula	:	MgAl_2O_4
Mol Wt.	:	142.27
Density	:	3.64 g/cm^3
Crystal forms	:	Cubic
Melting point	:	2135 °C
Specific gravity	:	3.3
Solubility	:	Insoluble in Water
Application	:	- structural, chemical, optical and electrical industry - uses as refractory in lining of steel making furnaces - water treatment and ceramics manufacturing

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Hydrothermal/Solvothermal Method

The word “*hydrothermal*” has geological origin. A self-explanatory word, “hydro” meaning water and “thermal” meaning heat. British Geologist, Sir Roderick Murchison (1792–1871) was the first to use this word, to describe the action of water at elevated temperature and pressure in bringing about changes in the earth’s crust leading to the formation of various rocks and minerals (Byrappa and Yoshimura, 2001).

In a sealed vessel (bomb, autoclave, etc.), solvents can be brought to temperatures well above their boiling points by the increase in autogenous pressures resulting from heating. Performing a chemical reaction under such conditions is referred to as solvothermal processing or, in the case of water as solvent, hydrothermal processing.

Materials and Methods

Magnesium aluminate (MgAl_2O_4) nanoparticles were prepared by hydrothermal assisted sol-gel method. 100 mL of magnesium chloride solution was added dropwise sodium hydroxide until pH 9 in 500 mL beaker, magnesium hydroxide precipitates were formed. And then 100 mL of aluminium nitrate solution mixed into the above solution. After mixing, added to 5 g of urea into this solution. Then the solution was transferred into a teflon-lined pressure vessel, which was sealed and heated on hotplate at 160 °C for 5 hours. After hydrothermal treatment, gel is formed. And then this gel was heated at sand bath, precursor powder were obtained. The resulted MgAl_2O_4 was calcinated at different temperatures of 600, 800, 1000, and 1150°C for 4 hours. The prepared MgAl_2O_4 was characterized by using TG-DTA, XRD, FT IR and FE-SEM techniques. Figure 1 shows photographs of MgAl_2O_4 nanoparticles prepared by hydrothermal assisted sol-gel method.

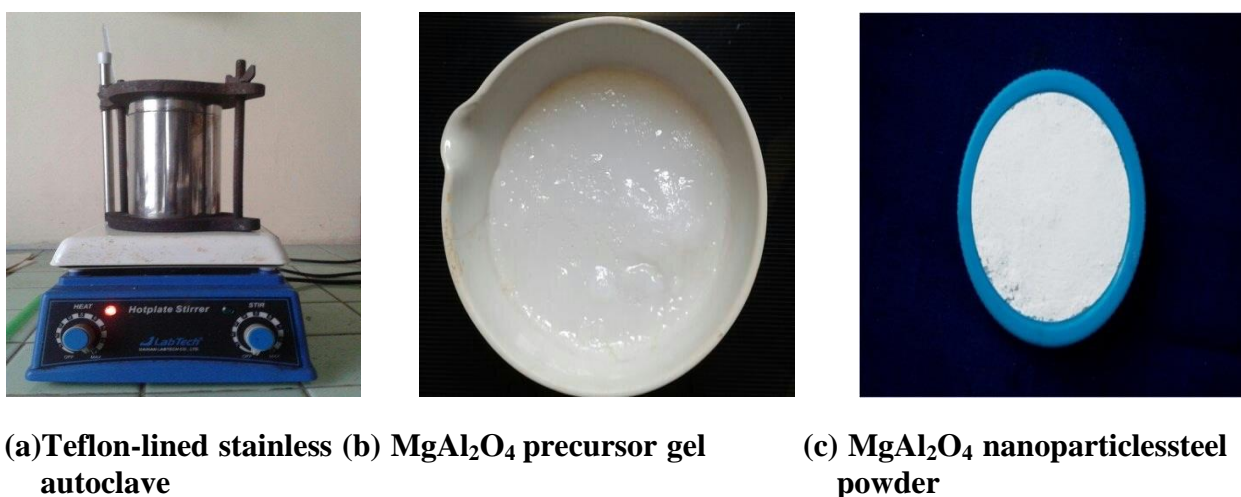


Figure 1. Photographs of MgAl_2O_4 nanoparticles prepared by hydrothermal assisted sol-gel method

Results & Discussion

TG-DTA thermogram of MgAl_2O_4

Thermal behavior of MgAl_2O_4 nanoparticles were determined by TG-DTA is shown in Figure 2. In TGA data, weight loss percent was found to be 17.69 %, which is caused by the dehydration of the sample. In DTA data, endothermic peak at 67°C is due to thermal effects of

volatile compound and absorbed water removal and at 317°C is related to the organic residues combustion.

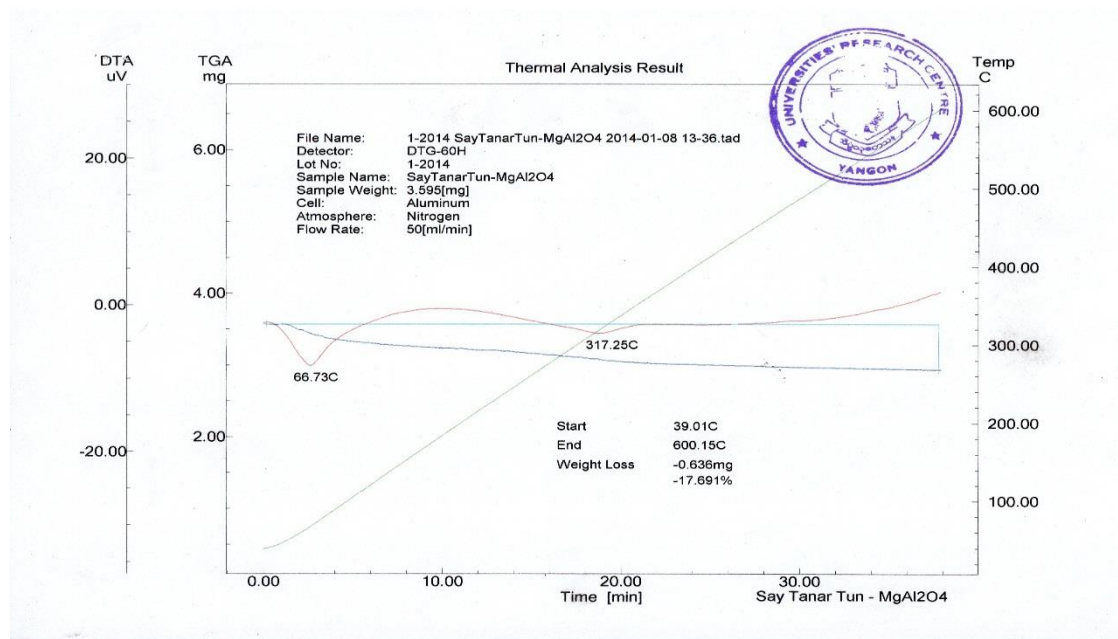


Figure 2. TG-DTA thermogram of MgAl_2O_4

XRD studies on calcinated products of MgAl_2O_4 nanoparticle

XRD patterns of the prepared spinel powder heat-treated at 600, 800, 1000 and 1150°C for 4 hours Figure 3. XRD data of the MgAl_2O_4 nanoparticles showed characteristic peaks related to Miller indices of 111, 220, 311, 400, 511, 440 and 531, these peaks are well matched with standard library data of JCPDS (75-1796>Spinel,syn- MgAl_2O_4). It was observed that the spinel powder was in amorphous nature up to 600°C. The spinel phase formation starts at 800°C and pure MgAl_2O_4 powder with found to be formed at 1000 and 1150°C. In Table 2, by using Scherrer equation, crystalline sizes of the MgAl_2O_4 nanoparticles obtained at calcination temperature of 800, 1000 and 1150°C were 10.15, 25.77 and 28.02 nm, respectively. The increase calcinations temperature yields the sharpness of the peaks, therefore crystalline size increased with increasing calcinations temperature and lattice constant 'a' decreased with increasing temperature shows in Table 3.

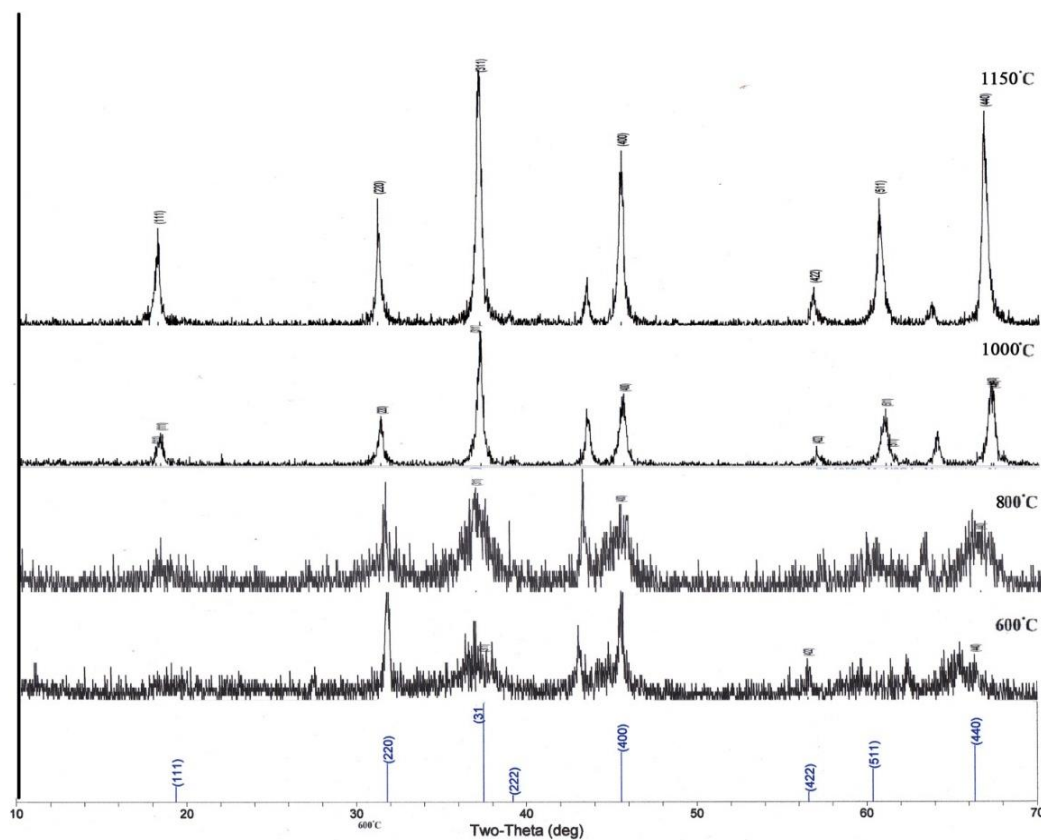


Figure 3. Changes of XRD pattern of MgAl_2O_4 with different calcination temperatures

Table 3. Crystallite Size and Lattice Constant of MgAl_2O_4 Nanoparticles at Different Calcination Temperatures

No.	Calcination Temperature (°C)	Crystallite size of MgAl_2O_4 (nm)	a (Lattice constant) (Å)
1	800	10.15	8.0658
2	1000	25.77	8.0607
3	1150	28.02	8.0328

Table 2. XRD Data of MgAl₂O₄ Nanoparticles (1150°C)

[MgAl2O4-DSTNTun.raw] MgAl2O4 - Full Chart											Peak Search Report
SCAN: 10.0/70.0/0.02/0.12(sec), Cu(40kV,40mA), I(max)=156, 03/19/14 18:40											
PEAK: 9-pts/Quartic Filter, Threshold=1.0, Cutoff=0.0%, BG=1/0.5, Peak-Top=Summit											
NOTE: Intensity = Counts, 2T(0)=0.0(deg), Wavelength to Compute d-Spacing = 1.54056Å (Cu/K-alpha1)											
#	2-Theta	d(Å)	(h k l)	BG	Height	Height%	Area	Area%	FWHM	XS(Å)	P/N
1	19.243	4.6087	(1 1 1)	5	60	39.5	14.6	29.6	0.225	399	3.6
2	31.463	2.8410	(2 2 0)	7	78	51.3	14.5	29.5	0.174	580	4.0
3	37.138	2.4189	(3 1 1)	12	152	100.0	49.3	100.0	0.299	297	5.7
4	45.061	2.0102	(4 0 0)	10	107	70.4	29.3	59.4	0.257	364	4.7
5	55.897	1.6435	(4 2 2)	2	24	15.8	6.1	12.3	0.234	425	2.2
6	59.520	1.5518	(5 1 1)	11	78	51.3	19.4	39.4	0.247	406	3.8
7	65.419	1.4254	(4 4 0)	13	131	86.2	36.8	74.7	0.265	384	5.2
8	68.858	1.3624	(5 3 1)	2	10	6.6	1.4	2.9	0.153	832	1.3

Scherrer equation

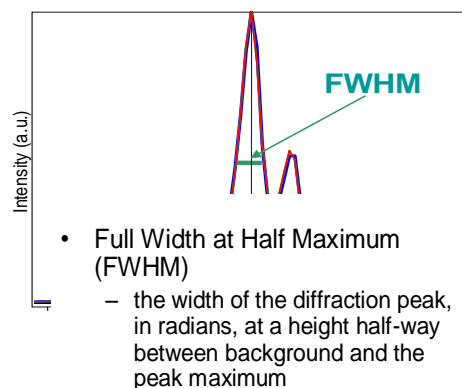
$$t = \frac{0.9\lambda}{B \cos \theta}$$

t = Crystallite size

 λ = Wavelength (Å) or nm θ = Diffraction angle of the peak under consideration at FWHM (°)

B= Observed FWHM (in radians)

t = 28.02 nm

**FT IR spectrum of MgAl₂O₄ nanoparticles**

The IR spectra of the calcined at 1000 and 1150 °C nanocrystalline powders in the wave number region from 4000 to 400 cm⁻¹ are shown in Figure 4. In the range of 3200-3700 is consistent with the O-H stretching of H₂O. The IR band provides evidence for the presence of organic residuals in the calcined powder in the wave number range of 1300-1400 and 1600-1700 cm⁻¹, which corresponds to C-O and C=C stretching vibrations, respectively. The visible band over the range of 1000-400 cm⁻¹ corresponds to metal-oxygen bonds (M-O-M).

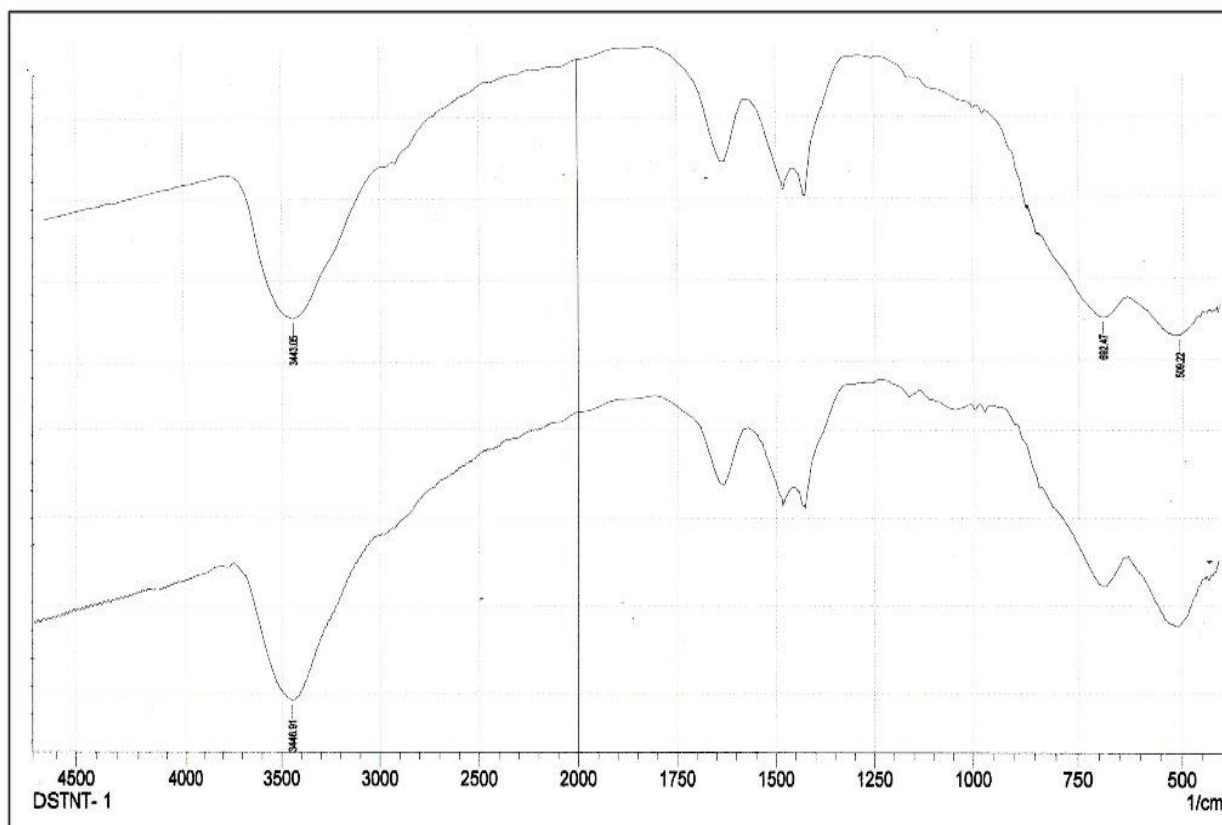


Figure 4. FT IR spectra of MgAl_2O_4 nanoparticles after calcination at 1000 and 1150 °C

FE-SEM micrograph of MgAl_2O_4 nanoparticles

The morphology of the precursors and nanoparticles was observed using field emission scanning electron microscopy (FE-SEM). In this work, SEM micrograph of MgAl_2O_4 nanoparticles obtained at 1150°C show in Figure 5 as flake-like shape.

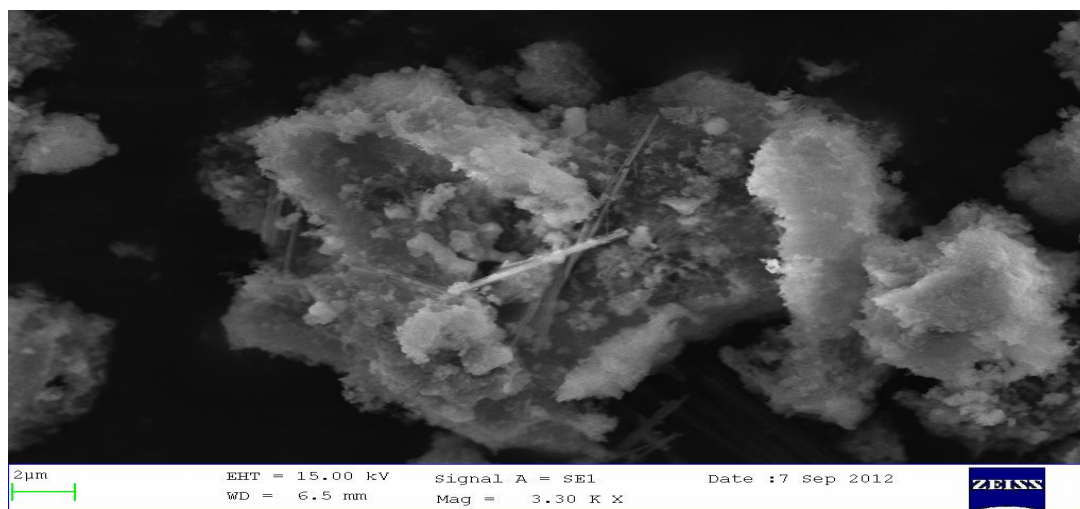


Figure 5. FE-SEM micrograph of MgAl_2O_4 nanoparticles

Conclusion

In this research, magnesium aluminate nanoparticles were synthesized by hydrothermal-assisted sol-gel method by using aluminium nitrate, magnesium chloride and urea. This method is simple, reliable and inexpensive method for the preparation of MgAl_2O_4 nanoparticles. The resulted magnesium aluminate powder was calcinated at different temperatures. XRD data of the MgAl_2O_4 nanoparticles showed characteristic peaks related to Miller indices of 111, 220, 311, 400, 422, 511, 440 and 531, these peaks are well matched with standard library data of JCPDS (75-1796>Spinel, syn- MgAl_2O_4). By using Scherrer equation, crystalline sizes of the MgAl_2O_4 nanoparticles obtained at calcination temperature of 800, 1000 and 1150 °C were 10.15, 25.77 and 28.02 nm, respectively. FT IR spectrum of MgAl_2O_4 nanoparticles were recorded and studied in the wave number range 400-4000 cm^{-1} . The band over the range of 1000-400 cm^{-1} corresponds to metal-oxygen bonds (Al-O and Mg-O). FE-SEM micrograph of MgAl_2O_4 nanoparticles showed as flake-like shape. Magnesium aluminate nanoparticles can be used in various industrial applications such as water treatment and ceramics manufacturing.

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