Preparation and Characterization of Magnesium Aluminate Nanoparticles by Hydrothermal Method
Saytanar Tun¹, Kyaw Naing²

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 MgAl₂O₄ 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- MgAl₂O₄). By using Scherrer equation, crystalline sizes of the MgAl₂O₄ nanoparticles obtained at calcination temperature of 800, 1000 and 1150°C were 10.15, 25.77 and 28.02 nm, respectively. FTIR spectra of MgAl₂O₄ 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 MgAl₂O₄ nanoparticles showed as flake-like shapes.

Keywords: MgAl₂O₄, 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 (MgAl₂O₄) 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 | MgAl₂O₄ |
| Mol Wt. | 142.27 |
| Density | 3.64g/cm³ |
| 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 |

¹ Assistant Lecturer, Department of Chemistry, Dagon University
² Deputy Permanent Secretary, Ministry of Education, Myanmar
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$_2$O$_4$) 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$_2$O$_4$ was calcinated at different temperatures of 600, 800, 1000, and 1150°C for 4 hours. The prepared MgAl$_2$O$_4$ was characterized by using TG-DTA, XRD, FT IR and FE-SEM techniques. Figure 1 shows photographs of MgAl$_2$O$_4$ nanoparticles prepared by hydrothermal assisted sol-gel method.

![Photographs of MgAl$_2$O$_4$ nanoparticles prepared by hydrothermal assisted sol-gel method](image)

**Figure 1.** Photographs of MgAl$_2$O$_4$ nanoparticles prepared by hydrothermal assisted sol-gel method

Results & Discussion

**TG-DTA thermogram of MgAl$_2$O$_4$**

Thermal behavior of MgAl$_2$O$_4$ 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$_2$O$_4$

XRD studies on calcinated products of MgAl$_2$O$_4$ nanoparticles

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$_2$O$_4$ 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$_2$O$_4$). 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$_2$O$_4$ powder with found to be formed at 1000 and 1150°C. In Table 2, by using Scherrer equation, crystalline sizes of the MgAl$_2$O$_4$ 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$_2$O$_4$ with different calcination temperatures

Table 3. Crystallite Size and Lattice Constant of MgAl$_2$O$_4$ Nanoparticles at Different Calcination Temperatures

<table>
<thead>
<tr>
<th>No.</th>
<th>Calcination Temperature ($^\circ$C)</th>
<th>Crystallite size of MgAl$_2$O$_4$ (nm)</th>
<th>a (Lattice constant) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>800</td>
<td>10.15</td>
<td>8.0658</td>
</tr>
<tr>
<td>2</td>
<td>1000</td>
<td>25.77</td>
<td>8.0607</td>
</tr>
<tr>
<td>3</td>
<td>1150</td>
<td>28.02</td>
<td>8.0328</td>
</tr>
</tbody>
</table>
Methods used in Jade to Define Peak Width

• Full Width at Half Maximum (FWHM)
  – the width of the diffraction peak, in radians, at a height half-way between background and the peak maximum

• Integral Breadth
  – the total area under the peak divided by the peak height
  – the width of a rectangle having the same area and the same height as the peak
  – requires very careful evaluation of the tails of the peak and the background

Scherrer equation

\[ t = \frac{0.9\lambda}{B\cos\theta} \]

\( t \) = Crystallite size
\( \lambda \) = Wavelength (Å) or nm
\( \theta \) = Diffraction angle of the peak under consideration at FWHM (°)
\( B \) = Observed FWHM (in radians)
\( t = 28.02 \) nm

FT IR spectrum of MgAl\(_2\)O\(_4\) 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 H\(_2\)O. 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).
The morphology of the precursors and nanoparticles was observed using field emission scanning electron microscopy (FE-SEM). In this work, SEM micrograph of MgAl$_2$O$_4$ nanoparticles obtained at 1150°C show in Figure 5 as flake-like shape.

Figure 4. FT IR spectra of MgAl$_2$O$_4$ nanoparticles after calcination at 1000 and 1150 °C

Figure 5. FE-SEM micrograph of MgAl$_2$O$_4$ 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 MgAl₂O₄ nanoparticles. The resulted magnesium aluminate powder was calcinated at different temperatures. XRD data of the MgAl₂O₄ 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- MgAl₂O₄). By using Scherrer equation, crystalline sizes of the MgAl₂O₄ 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 MgAl₂O₄ 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 MgAl₂O₄ 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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References