Morphological Characterizations of Spinel MgAl$_{2-x}$Fe$_x$O$_4$ Nanoparticles Synthesized by Sol-Gel Method

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Abstract. In this paper, pure magnesium alumina (MgAl$_2$O$_4$) and magnesium alumina spinel nanoparticles doped iron (MgAl$_{2-x}$Fe$_x$O$_4$) where (X= 0.01, 0.02, 0.03, 0.04, 0.05) were by sol-gel method. The calcination operation was performed at a temperature of 800 °C at a rate of 5 °C / min. The resulting materials of MgAl$_{2-x}$Fe$_x$O$_4$ nanoparticles were determined using XRD, FT-IR, EDX, SEM methods. XRD results show that MgAl$_2$O$_4$ and Fe$^{3+}$; MgAl$_{2-x}$O$_4$ nanoparticles have a single-phase at 800 °C. FTIR analyze confirmed the presence of MgAl$_2$O$_4$ and Fe$^{3+}$; MgAl$_{2-x}$O$_4$ as well as FTIR analysis shows its composition and structure. Surface morphology examinations by scanning electron microscopy (SEM) show that the nanoparticles synthesized in this way are estimated to be angular, uniform and about 50-70 nm in size.

Keywords: Magnesium aluminate spinel, Iron, Dopind, Sol-gel

1. Introduction
Spinal MgAl$_2$O$_4$ is one of the oxides that has special properties that include high melting point (2135 °C), low thermal expansion coefficient, low dielectric drop and excellent resistance to acid and base [1]. In these structures, oxygen is located in the FCC. The cation (Mg) is in the quadrilateral intermediate position and the cation (Al) is in the octaver intermediate position [1-3]. Due to spinel structure of MgAl$_2$O$_4$ its special properties such as high melting point in refractory, humidity sensors and catalyst bases [4]. Because of its good radiation resistance, it is suitable for use in electrical insulation components for fusion reactors [5-6] by adding metal ions and rare ions [7]. Its general form is as : ($Mg_{1-2x}Al_{2x}$)($Mg_{2x}Al_{2-2x}$)O$_4$. If λ=0, the spinel is normal and if it is λ=1/2 , it is an inverted spinel, and for the values between the two, it is called a spinel with a random cation distribution. The interaction potential between lattice ions includes Coulomb potential and short-range repulsion, and will vary depending on the type of spinel, the amount of energy, the lattice constant, and the bulk modulus [8].pure and doped MgAl$_2$O$_4$ spinel is synthesized by different methods such as decomposition of plasma spray oxides, fuel synthesis, whose equipment and raw materials are expensive[9].

In conventional methods for preparation nano-materials, powders become contaminated due to grinding, and some of their properties change. The sol-gel method to have a homogeneous product with a high degree of purity. The synthesis of sol-gel uses hydrolyzable salts that can be dissolved in water and alcohol. This definition is often extended to any system such as colloidal cells and soluble
salts that undergo sol gel evolution, so we use aluminum, magnesium, and iron salts in synthesis by sol gel method [10].

There is a group of scientific research and studies that dealt with the issue of preparing (MgAl2O4) spinel in many ways, including the Sol-gel method that begins with the source [11-17]. As well as other scientific studies that dealt with the preparation and manufacture of doping spinel (MgAl2-xR2O4), Where (R=Fe3+, Ce4+, Mn3+, ... etc), which begins with the source [18-25].

The advantages of the sol-gel method include high purity of nanoparticles produced, reduction of sintering temperature, use of cheap raw materials, non-toxicity and no need for complex and expensive equipment and devices [26]. In this study, magnesium aluminate spinel nanoparticles pure and doped were prepared by sol-gel method and then using XRD, scanning electron microscopy (SEM) with the energy dispersive x-ray spectroscopy (Edx) and Fourier transform red spectrum (FT-IR) were examined.

2. Materials and research methods

2-1 Synthesis of magnesium aluminate spinel:
In the Preparation of spinel MgAl2O4, the modified sol-gel method was used. From hydrated magnesium nitrate Mg(NO3)2.6H2O, hydrated aluminum nitrate Al(NO3)3.6H2O, water H2O and anhydrous citric acid C6H8O7 were used as raw materials in this synthesis. All the consumables were (THOMAS BAKER) type as shown in Table 1.

The reaction's general chemical equation can be written as follows, with all undesirable by-products such as (CO2, H2O, NOx), and so on written as "Z"

\[ Mg(NO_3)_2 \cdot 6H_2O + 2Al(NO_3)_3 \cdot 9H_2O + 5C_6H_8O_7 \rightarrow MgAl_2O_4 + Z \]

2-1-1 Experimental procedure:
To prepare ofMgAl2O4 spinel precursor dissolve (1.80 g) Mg(NO3)2.6H2O in (10 ml) of distilled water and slowly dissolve (6.75 g) of anhydrous citric acid (C6H8O7) add the solution and, while stirring the solution with a magnetic stirrer, heat it at 60°C for one hour, until the solution is completely homogeneous, then (5.27 g) Al(NO3)3.6H2O dissolve in (15 ml) of distilled water and add to the solution, at this point the solution is heated to 80°C for one hour. The color of the solution after the addition of hydrated aluminum nitrate turns milky, with further heating of the solution to 140 °C for one hour, the residue solvent evaporates, the solution becomes viscous and finally turns into a xerogel, to complete drying and removal the organic solvent is placed in a relatively dry xerogel in an oven at 200 °C for 1 hours. In this case, a dark yellow powder is obtained as the precursor. Than calcinated to 800 °C in a furnace at 5 °C/min. This step is called calcination of powder, which is a common heat treatment in the final preparation of nano crystalline powders. The powder obtained from this step is white. The steps in the preparation of magnesium aluminate (MgAl2O4) spinel nanoparticles are summarized in Figure 1.

![Figure 1. Preparation steps of MgAl2O4 Nanoparticles](image-url)
2-2 Synthesis of iron (III)-doped magnesium aluminate spinel:
In preparing the powder (MgAl$_{2-x}$Fe$_x$O$_4$) in addition to the consumables mentioned in the first stage, water was used as a solvent for preparing sol. Multiple quantities of iron nitrate (III), represented by the symbol "X", were added, where x= (0.01, 0.02, 0.03, 0.04, 0.05) to the solution and it is a solution of colored bricks. According to stoichiometric relationships the chemical formula in this case is Mg(Al$_{2-x}$Fe$_x$)O$_4$. The gel was prepared and placed in a dryer at 150 °C to obtain a crude powder, and finally, calcinate to obtain the final powder, by calcination at a temperature of 800 °C at a rate of 5 °C / min.
The rest of the steps are the same as preparing the MgAl$_2$O$_4$ powder spinel, with the exception that the synthesized powder is not white. However, different colors appear according to the relative proportions. All the consumables were (THOMAS BAKER) type as shown in Table 1.

| Name of the material            | Chemical formula | Molecular Weight (g/mol) | purity | Company          |
|---------------------------------|------------------|--------------------------|--------|------------------|
| hydrated magnesium nitrate      | Mg(NO$_3$)$_2$.6$H_2$O | 256.41                   | 98%    | THOMAS BAKER     |
| Hydrated Aluminum Nitrate       | Al(NO$_3$)$_3$.6$H_2$O | 375.13                   | 98%    | THOMAS BAKER     |
| Hydrated Iron(III) Nitrate      | Al(NO$_3$)$_3$.6$H_2$O | 404.00                   | 98%    | THOMAS BAKER     |
| anhydrous citric acid           | C$_6$H$_8$O$_7$   | 192.12                   | 99%    | THOMAS BAKER     |
| Water                           | $H_2$O           | 18.0153                  | 99%    | -                |

Table 1. Raw materials used in the synthesis of Spinel MgAl$_{2-x}$Fe$_x$O$_4$ nanopowders by Sol-gel method

3. Results and Discussion

3.1 XRD of magnesium aluminate spinel and Nanoparticles:
The X-ray diffraction pattern for MgAl$_2$O$_4$ spinel nanoparticles calcined at 800°C is shown in Figure (2). The results of the XRD pattern indicate that The MgAl$_2$O$_4$ spinel is completely pure, with a cubic and a single-phase spinel formed at a temperature of 800 °C. It can be observed that ultrafine spinel phase (MgAl$_2$O$_4$) card-No. (96-900-7136) with crystallite size of (10.81nm) were obtain at a temperature of 800 °C. this mean that the clear gel transformation ultrafine powder of MgAl$_2$O$_4$ spinel. Crystalline intensity peaks of MA (MgAl$_2$O$_4$) spinel appeared at (20) value (18.86, 31.16,44.79, 55.46, 59.25, 65.03, 66.49). The peaks are responding to (111), (220), (311), (400), (511) and (444) diffraction plans respectively agreement with [27]. MA phase is identified as a cubic structure with space group; Fd3m. The crystallite size of the formed MA spinel nanopowders was calculated from XRD analysis using Debye-Scherer formula of the most intense peaks (311) given in Equation (1). An estimate measured that:

$$D=K\lambda/(\beta \cos \theta) \quad ...........\text{Eq}(1)$$

In this formula, (β) is half the width of the maximum peak height, (K) is a fixed number, about 0.9, (λ) is the X-ray wavelength that is read from the device, and θ is the angle that is read from the horizontal axis XRD.
The diffraction patterns (XRD) for MA spinel doped with in iron (III) are shown in Figure 2. Peaks indicate that a single-phase spinel has been obtained. For more certainty, the standard pattern is given
in section c) of Figure 2. Therefore, both samples are fully formed at 800 ° C. Also, the network constant for the structure of \( MgAl_{2-x}Fe_xO_4 \)'s was calculated using XRD data of 8.083Å and 8.109Å, respectively, which is very close to the value written in the reference books [28]. From Figure 2, using the Debye-Scherrer formula, the particle size of prepared by this method was calculated and given in table 2.

**Table 2**. Crystallite size (nm) of Spinel \( MgAl_{2-x}Fe_xO_4 \) nanopowders by Sol-gel method at calcination temperature 800 °C.

| Sample                  | Average crystallite size (nm) |
|-------------------------|------------------------------|
| \( MgAl_2O_4 \)         | 10.81                        |
| \( MgAl_{2-0.01}Fe_{1.99}O_4 \) | 11.68                        |
| \( MgAl_{2-0.02}Fe_{1.99}O_4 \) | 10.80                        |
| \( MgAl_{2-0.03}Fe_{1.97}O_4 \) | 13.36                        |
| \( MgAl_{2-0.04}Fe_{1.96}O_4 \) | 12.35                        |
| \( MgAl_{2-0.05}Fe_{1.95}O_4 \) | 9.14                         |

![Images of XRD patterns for different samples](image)
3.2. FT-IR spectrum of MgAl2O4 and \( Mg(Al_{2-x}Fe_x)O_4 \) Nano particles:

Figure 3 shows the FT-IR spectrum of \( MgAl_2O_4 \) nanoparticles, with curve (a) corresponding to pre-calcination and curve (b) corresponding to calcination of the precursor. As can be seen in curve a (before calcination), the intensity of the adsorption peaks due to the formation of organic bonds (related to citric acid and water) is very high. By analyzing the b-curve spectrum (corresponding to calcined powder at 800 °C), there is no peak that can be considered to belong to organic groups in the spectrum, the existing precursor is completely disappear by calcination at 800 °C to give pure nanopITICAL. The absorption peak in the range of 531-690 cm\(^{-1}\) in the FT-IR spectrum obtained from the synthesized material is related to the Al-O-Mg bonds, which indicates the formation of \( MgAl_2O_4 \) spinel nanoparticles agreement with [29]. Investigating FTIR spectra with increasing percentage of additives \((x=0.01,0.02,0.03,0.04,0.05)\), the polymer structure is destroyed and it can be said that there is almost no organic bond at 800 °C. In the spectrum corresponding to the spinel \( MgAl_2O_4 \) and spinel \( Fe^{+3} \) \( MgAl_2O_4 \), the O-H bonds and the peaks bound to the organic bonds are very low in intensity and the adsorption density appears to be due to an increase in inorganic bonds. The links have become stronger. This achievement confirms the results of X-ray diffraction, In the wave number between 400 to 1000 cm\(^{-1}\), the absorption related to Al-O-Mg bonds appears, in other words, the peak 531-690 cm\(^{-1}\) corresponds to the spinel \( MgAl_2O_4 \). After iron doping, the peak intensity of the Al bond decreases and the peak intensity of the Fe bond increases, which indicates the replacement of iron with aluminium, or due to the fact that in the MgAl204 spinel, trivalent ions are placed in Octahedral positions. iron ion will be replaced in empty Octahedral positions. (Figure 3).
Figure 3. FTIR spectra of $MgAl_{1.99}Fe_{0.01}O_4$ and $MgAl_{1.98}Fe_{0.02}O_4$ nanoparticles calcined at 800 °C.
3.3. SEM results of $\text{MgAl}_2\text{O}_4$ spinel and $\text{MgAl}_{2-x}\text{Fe}_x\text{O}_4$ nanoparticles:

Surface morphology of $\text{MgAl}_2\text{O}_4$ spinel nanoparticles and iron doped samples with iron are shown in Figure (4, 6, 8) respectively. From Figure (4), the $\text{MgAl}_2\text{O}_4$ particles form completely porous and angular structures that are perfectly bonded together and have a size on the nanometer scale. In Figure (6, 8), Spinel is morphologically similar in both cases, and in both cases the appearance of the particles is "angular". The particle size is estimated to be in the range of 50-70 nm. An example of an element analysis performed by the EDX method is shown in Figure (5). At SEM micrographs, the EDX provides the type and weight percent of each element present in the selected point of sample agreement with [30]. We can notice the differences between EDX analysis of pure and doped $\text{MgAl}_2\text{O}_4$, which resulted in the increase in the percentage of O, Fe and decrease in the percentage of Al. Furthermore, EDX analysis revealed that the highest ratio of Oxygen atom was 57.52% at (0.5) this directly suggests that the introduction of iron was at the expense of oxygen. The results of EDX analysis show that the amount of Mg and Al in the resulting powder compared to their load, not only do not differ much but also have a very good compatibility with each other. Table 3 shows the Mg and Al levels from the EDX results and Table 4, 5 shows the Mg, Al and Fe levels from the EDX results.

![Figure 4](image_url)  
**Figure 4.** SEM image of $\text{MgAl}_2\text{O}_4$ powder in four different magnifications.
Table 3. Mg and Al levels from the EDX analysis.

| Element | Series | Wt.% | At.% |
|---------|--------|------|------|
| Mg      | K-series | 16.25 | 13.29 |
| Al      | K-series | 34.38 | 25.34 |
| O       | K-series | 49.38 | 61.37 |

Figure 5. EDX analysis of spinel nanoparticles MgAl₂O₄ calcined at 800 °C.
Figure 6. SEM image of $\text{MgAl}_{1.97}\text{Fe}_{0.03}\text{O}_4$ powder in four different magnifications.

Table 4. Mg, Al and Fe levels from the EDX analysis.

| Element | Series  | Wt.%  | At.%  |
|---------|---------|-------|-------|
| Mg      | K-series | 16.36 | 13.46 |
| Al      | K-series | 33.55 | 24.87 |
| Fe      | K-series | 1.07  | 0.38  |
| O       | K-series | 49.03 | 61.29 |

Figure 7. EDX analysis of spinel nanoparticles $\text{MgAl}_{1.97}\text{Fe}_{0.03}\text{O}_4$ calcined at 800 °C.
Figure 8. SEM image of $MgAl_{1.95}Fe_{0.05}O_4$ powder in four different magnifications.

Table 5. Mg, Al and Fe levels from the EDX analysis.

| Element | Series | Wt.% | At.% |
|---------|--------|------|------|
| Mg      | K-series | 17.09 | 14.48 |
| Al      | K-series | 35.29 | 26.93 |
| Fe      | K-series | 2.93  | 1.08  |
| O       | K-series | 44.69 | 57.52 |
Figure 9. EDX analysis of spinel nanoparticles $MgAl_{1.95}Fe_{0.05}O_4$ calcined at 800 °C

4. Conclusion:

- The sol-gel is a very useful technique in the preparation of pure and doped MgAl$_2$O$_4$ since the X-rays confirmed by the normal data and SEM images with the energy dispersive x-ray spectroscopy (Edx) as well as the FTIR images. The second explanation is that the Sol-Gel method offers materials of nanosizing.
- According to the different methods and raw materials used for the synthesis of MgAl$_2$O$_4$ and Fe$^{3+}$:MgAl$_2$O$_4$, in the sol-gel method, the samples were formed at a temperature of 800 °C.
- The SEM images taken from the powders show it has dimensions of about nanometers and has the appearance of angular particles.
- The results of EDX analysis show that the amount of Mg, O and Al in the resulting powder have been confirm purity.
- The peaks appearing in the range of 531-690 cm$^{-1}$ in the FTIR spectrum obtained from the synthesized material, indicate the formation of MgAl$_2$O$_4$ spinel. The structural shifts because of changes in the value of x (iron content) have affected the grid vibrations, so Fig.3 shows that there is a minor adjustment of the maximum positions of x. The lattice vibrations were also affected by the cation mass and bonding power.
- Since the ionic radius of Fe is greater than Al, and as can be seen from the Bragg relation ($2d\sin\theta = n\lambda$), as d becomes larger, the angle $\theta$ must become smaller, which is due to the X-ray diffraction pattern and XRD spectrum displacement. Relevant, Fe$^{3+}$:MgAl$_2$O$_4$ relative to the pure spinel MgAl$_2$O$_4$ indicates that the iron ion has replaced the aluminium ion or has been replaced in the octahedral voids.
- The use of sol-gel method results in homogeneous and high purity products. The temperature of the heat treatment is low. This method leaves little waste than other methods.
References:

1. Kingery, W. D., Bowen, H. K., & Uhlmann, D. R. (1976). Introduction to ceramics (Vol. 17). John wiley & sons.
2. Saleem, M., Padole, M., & Mishra, A. (2019). Low dielectric constant and signature of ferroelectric nature in transition metal (Co, Ni, Cu)-doped Mg 0. 5 Zn 0. 5 Al2O4 aluminates. Journal of Advanced Dielectrics, 9(04), 1950034.
3. Ganesh, I. (2013). A review on magnesium aluminate (MgAl2O4) spinel: synthesis, processing and applications. International Materials Reviews, 58(2), 63-112.
4. MCENTIRE, B., & Pressing, D. (1991). Engineering Materials Handbook, Vol. 4-Ceramic and Glasses, ASM International. The Materials Information Society.
5. Salomes, J., Galicia, J. A., Wang, J. A., Valenzuela, M. A., & Aguilar-Rios, G. (2000). Synthesis and characterization of nanocrystallite MgAl2O4 spinels as catalysts support. Journal of materials science letters, 19(12), 1033-1037.
6. Anghel, S. (2011). Optical processes in α-ZnAl2S4 spinel-type single crystals doped by transition metals ions: ti, Co et V (Doctoral dissertation, Université Claude Bernard-Lyon I).
7. Ganesh, I., Johnson, R., Rao, G. V. N., Mahajan, Y. R., Madavendra, S. S., & Reddy, B. M. (2005). Microwave-assisted combustion synthesis of nanocrystalline MgAl2O4 spinel powder. Ceramics International, 31(1), 67-74.
8. MCENTIRE, B., & Pressing, D. (1991). Engineering Materials Handbook, Vol. 4-Ceramic and Glasses, ASM International. The Materials Information Society.
9. Koch, C. C. (2006). Nanostructured materials: processing, properties and applications. William Andrew.
10. Scherer, G. W. (1989). Drying gels: VIII. Revision and review. Journal of non-crystalline solids, 109(2-3), 171-182.
11. Alvar, E. N., Rezaei, M., & Alvar, H. N. (2010). Synthesis of mesoporous nanocrystalline MgAl2O4 spinel via surfactant assisted precipitation route. Powder Technology, 198(2), 275-278.
12. Hsu, C. H., Lin, J. S., & Yang, H. W. (2011). Fabrication and characterization of MgAl2O4 thin films by sol-gel method. In Advanced Materials Research (Vol. 216, pp. 514-517). Trans Tech Publications Ltd.
13. Das, R. R., Nayak, B. B., Adak, S., & Chattopadhyay, A. K. (2012). Influence of nanocrystalline MgAl2O4 spinel addition on the properties of MgO-C refractories. Materials and Manufacturing Processes, 27(3), 242-246.
14. Nassar, M. Y., Ahmed, I. S., & Samir, I. (2014). A novel synthetic route for magnesium aluminate (MgAl2O4) nanoparticles using sol–gel auto combustion method and their photocatalytic properties. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 131, 329-334.
15. Balabanov, S. S., Yavetskiy, R. P., Belyaev, A. V., Gavrishchuk, E. M., Drobotenko, V. V., Evodokimov, I. I., ... & Pimenov, V. G. (2015). Fabrication of transparent MgAl2O4 ceramics by hot-pressing of sol-gel-derived nanopowders. Ceramics International, 41(10), 13366-13371.
16. Habibi, N., Wang, Y., Arandiyan, H., & Rezaei, M. (2017). Low-temperature synthesis of mesoporous nanocrystalline magnesium aluminate (MgAl2O4) spinel with high surface area using a novel modified sol-gel method. Advanced Powder Technology, 28(4), 1249-1257.
17. Shahbazi, H., & Tataei, M. (2019). A novel technique of gel-casting for producing dense ceramics of spinel (MgAl2O4). Ceramics International, 45(7), 8727-8733.
18. Hassanzadeh-Tabrizi, S. A. (2011). Polymer-assisted synthesis and luminescence properties of MgAl2O4: Tb nanopowder. Optical Materials, 33(11), 1607-1609.
19. Raj, S. S., Gupta, S. K., Grover, V., Muthe, K. P., Natarajan, V., & Tyagi, A. K. (2015). MgAl2O4 spinel: Synthesis, carbon incorporation and defect-induced luminescence. Journal of Molecular Structure, 1089, 81-85.

20. Kolesnikov, I. E., Golyeva, E. V., Kurochkin, A. V., & Mikhailov, M. D. (2016). Structural and luminescence properties of MgAl2O4: Eu3+ nanopowders. Journal of Alloys and Compounds, 654, 32-38.

21. Motloung, S. V., Dejene, B. F., Kroon, R. E., Ntwaeaborwa, O. M., Swart, H. C., & Motaung, T. E. (2017). The influence of Cr3+ concentration on the structure and photoluminescence of MgAl2O4: 0.1% Eu3+, x% Cr3+ (0≤ x≤ 0.15%) nanophosphor synthesized by sol-gel process. Optik, 131, 705-712.

22. Menon, S. G., Hebbar, D., Choudhari, K. S., Santhosh, C., & Kulkarni, S. D. (2019). Effect of Zn substitution in Cr3+ doped MgAl2O4 mixed spinel nanoparticles on red/NIR emission properties. Materials Research Bulletin, 111, 294-300.

23. Basyrova, L., Balabanov, S., Belyaev, A., Drobotenko, V., Volokitina, A., Vitkin, V., ... & Loiko, P. (2019, December). Synthesis, structure and spectroscopy of Fe2+: MgAl2O4 transparent ceramics. In Journal of Physics: Conference Series (Vol. 1410, No. 1, p. 012123). IOP Publishing.

24. Pribyl, R., Stastny, P., Pazderka, M., Kelar, J., Tucekova, Z. K., Zemanek, M., ... & Cernak, M. (2020). Properties of MgAl2O4 doped alumina barrier layers for dielectric barrier discharge. Journal of Physics D: Applied Physics, 53(50), 505202.

25. Golyeva, E. V., Vaishlia, E. I., Kurochkin, M. A., Kolesnikov, E. Y., Lähderanta, E., Semencha, A. V., & Kolesnikov, I. E. (2020). Nd3+ concentration effect on luminescent properties of MgAl2O4 nanopowders synthesized by modified Pechini method. Journal of Solid State Chemistry, 289, 121486.

26. Sharma, M., Pathak, M., & Kapoor, P. N. (2018). The sol-gel method: pathway to ultrapure and homogeneous mixed metal oxide nanoparticles. Asian J. Chem., 30(7), 1405-1412.

27. Ewais, E. M., El-Amir, A. A., Besisa, D. H., Esmat, M., & El-Anadouli, B. E. (2017). Synthesis of nanocrystalline MgO/MgAl2O4 spinel powders from industrial wastes. Journal of Alloys and Compounds, 691, 822-833.

28. Wilkerson, K. R. (2012). Thermal properties of spinel based solid solutions.

29. Sanjabi, S., & Obeydavi, A. (2015). Synthesis and characterization of nanocrystalline MgAl2O4 spinel via modified sol–gel method. Journal of Alloys and Compounds, 645, 535-540.

30. Li, H., Wei, H. Y., Cui, Y., Sang, R. L., Bu, J. L., Wei, Y. N., ... & Zhao, J. H. (2017). Synthesis and characterization of MgAl2O4 spinel nanopowders via nonhydrolytic sol–gel route. Journal of the Ceramic Society of Japan, 125(3), 100-104.