



A Study on the Morphology and Physical-Mechanical Properties of Low-Density Spinel Fabrication

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Abstract

As the Nigerian defence industry grows, spinel has the potential to become the next material for advanced military applications. This will reduce the over-reliance on imports. It is a critical material for bulletproof windows for armoured vehicles, personal protective gear, missile domes and night vision systems. Magnesium aluminate Spinel (MAS) is a face-centred cubic synthetic material with excellent, chemical, mechanical, optical, thermal, and dielectric properties. Slip casting methodology was adopted and the samples (A, B, C, D) were drawn and analysed for mechanical tests and characterization of the constituent elements, through XRD and SEM analyses. The results of the XRD analysis for all samples were at high intensity at $2\theta=65.42^\circ, 65.56^\circ, 59.92^\circ$ where spinel was formed. The SEM micrographs show the low dense characteristics of the spinel formation which was the highest with 68% observed in sample D. The mechanical test results showed that sample B recorded the highest flexural strength of 210 MPa, followed by Sample D with 150 MPa and sample C with 145MPa. In conclusion, low-density spinel production was achieved, however for a more densified output, the study suggests adopting a pressured sintering approach.

Keywords: Spinel, fabrication, slip casting, armour, characterisation, ceramics

1. Introduction

The Nigerian State is facing significant security challenges both domestically and internationally, including issues like banditry, kidnapping, terrorism, and ongoing conflicts between farmers and herders (Abdullahi & Mukhtar, 2022). In response to these national security threats, Nigeria has allocated more than 4.76 billion USD for defence, which represents 22.4% of global security spending (Ayeni, 2024; Minini & Selem-Amachree, 2020). To reduce this high expenditure, this study aims to explore the development of locally produced armor materials that can be used in the manufacturing of various strategic security resources.

Spinel is a stable binary compound within the $\text{MgO-Al}_2\text{O}_3$ system, belonging to a group that includes various oxides. Its structural formula is AB_2O_4 , where A represents a divalent metallic ion like Mg^{2+} or Ni^{2+} , and B denotes a trivalent metallic ion, such as Al^{3+} or Cr^{3+} (Wang et al., 2015). A unique feature of spinel is its capacity for its components to be substituted with other small ions (Bowles, 2021; Klym et al., 2014).

The crystal structure of spinel was uniquely and independently established by Bragg and Nishikawa. Four primitive tetrahedral unit cells come together to create the standard cubic unit cell of spinel. Each unit cell contains $z=8$ formula units, which are made up of 32 anions and 24 cations, resulting in a total of 56 atoms (Sickafus et al., 1999). Different distributions of cations in spinel indicate varying ratios of A and B ions occupying the tetrahedral and octahedral sites. Spinel can be categorized into three types: normal, inverse, and complex spinel. These types are defined by the formula $A_{1-\lambda}B_\lambda(A_\lambda B_{2-\lambda})X_4$. In this notation, the ions

before the parentheses are situated in the tetrahedral sites, while those within the parentheses are found in the octahedral sites. A value of $\lambda=0$ corresponds to "normal" spinel, $\lambda=1$ corresponds to "inverse" spinel, and values of $0<\lambda<1$ indicate "complex" spinel (Sickafus et al., 1999; Zhao et al., 2017).

As the temperature rises, the area of the MgAl_2O_4 phase expands significantly, resulting in a higher concentration of alumina. This material exhibits outstanding performance at high temperatures, a low thermal expansion rate, strong thermal shock resistance, and excellent corrosion resistance. Consequently, it is primarily utilized in various industrial applications, including open steel furnaces, active link kilns, ladles, and cement rotary kilns (Wang et al., 2015). MgO occurs in metamorphosed limestones and dolomites, exhibiting perfect cleavage along the [001] direction and belonging to the cubic space group known as periclase. This mineral is formed through the heating of dolomite and is primarily utilized as a refractory material. The color of periclase can range from white to yellow or brown, depending on the presence of iron (Fe). When trace amounts of chromium (Cr) and iron are included in MgO , green periclase is produced, while magnesium-zinc combinations yield grass-green periclase (Li et al., 2008). Aluminum oxide (Al_2O_3) crystallizes into corundum, which features a trigonal structure. The red variant of corundum is referred to as ruby, while the blue variant is known as sapphire. Corundum can be found in metamorphic rocks rich in aluminum and exists in both natural and synthetic forms. The various colors of corundum, including red, blue, green, yellow, orange, pink, and brown, arise from different transition stages between corundum and aluminum hydroxides. In contrast, chromates exhibit a cubic structure, classified under space group $\text{Fd}\bar{3}\text{m}$ (No. 227) in the International Table (International Union of Crystallography, 1952). Similar to other members of the spinel group, it is created from a nearly cubic close-packed arrangement of oxygen atoms, with an aluminum atom coordinated in an octahedral structure (Burns & Glazer, 2013). Spinel is primarily manufactured using two main approaches: solid-state reaction methods and wet chemical methods. The solid-state methods encompass techniques such as sintering, electric melting, molten salt processing, and mechanical synthesis, which are commonly utilized for industrial mass production of refractories. On the other hand, the wet chemical methods include co-precipitation, combustion synthesis, and hydrothermal synthesis, which are employed to create optically transparent ceramic materials, catalysts, catalyst carriers, and humidity sensors (Qiao & Wen, 2020). The conventional method for synthesizing spinel has historically involved a high-temperature solid-state process (Butt & Rehman, 2019). However, because this approach requires significant energy and complex equipment, there is a need to adopt newer techniques to produce spinel with various sizes and shapes (Sickafus et al., 1999; Butt & Rehman, 2019). The creation of spinel is a complex, costly, and time-intensive process. Therefore, this study focused on developing a more affordable spinel and examining its unique properties.

2. Materials and Methods

2.1. Materials:

Magnesium Oxide (MgO) powder, gypsum (plaster of Paris) and Alumina were purchased from the available market.

2.2. Methods:

2.2.1. Spinel Manufacturing

The spinel samples were produced through slip casting, a process that entails mixing alumina and magnesia with sintering aids to create a slurry. This mixture was stirred manually for 5 minutes until all bubbles disappeared. The slurries were then poured into molds and allowed to settle. After this settling period, green ceramics were formed and subsequently dried at room temperature (Nurudeen et al., 2024).

2.2.2. Mould formation:

The mold for slip casting was created using a plaster-to-water ratio of 3:2, resulting in a plaster block measuring 130 x 25 mm³. Figure 1 illustrates the mold utilised for the production of spinel. Slip-casting experiments were conducted according to the quantities specified in the design array presented in Table 1.



Fig. 1: Moulds for Flexural Strength Samples

Table 1: Sample Design for Spinel

Constituent mass (g)	A	B	C	D
Mg	10	15	20	25
TiO ₂	2	4	6	8
Al ₂ O ₃	25	50	75	100

Table 1 outlines the sample design for specimens A-D. The selection criteria were informed by earlier research, which suggested that the magnesium content should not surpass 30% by weight, as this can affect both density and porosity. (Nurudeen et al., 2024; Ghosh et al., 2004).

2.3. Mechanical Testing:

Flexural tests were conducted using a Three-point Flexural Testing Machine shown in Figure 2; measurements were done according to ASTM D3039 (Nurudeen et al., 2024).

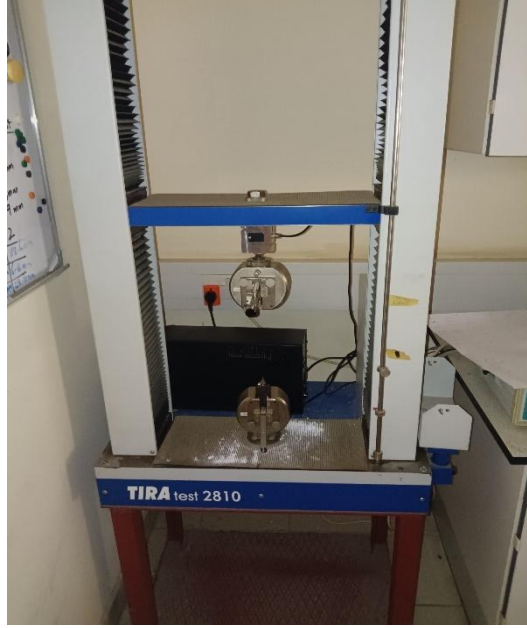


Fig. 1: Flexural Testing Machine

2.4. Scanning Electron Microscopy (SEM):

The scanning was performed using an EDX scanning electron microscope (SEM PRO X) to measure the SEM micrographs at different magnification levels (Nurudeen et al., 2024; Sinhamahapatra et al., 2022).

2.5. X-Ray Diffraction (XRD)

RIGAKU MINI FLEX XRD machine was used according to (Carter & Norton, 2013; Brundle et al., 1992).

2.6. Sintering Process

The green ceramics were arranged in the furnace and then sintered at 1600°C using Vecstar Muffle Furnace and Carbolite HTF with a maximum capacity of 1800°C. The sintering temperature of raw MgO content on the formation of spinel adopted was 1600 °C for 2h through a single stage firing process (Sarkar et al., 2004). Sintering aid (TiO_2) was used in reducing the sintering time and accelerate phase formation and densification (Ganesh, 2013). Figure 3 shows the flexural test samples after being cooled overnight.

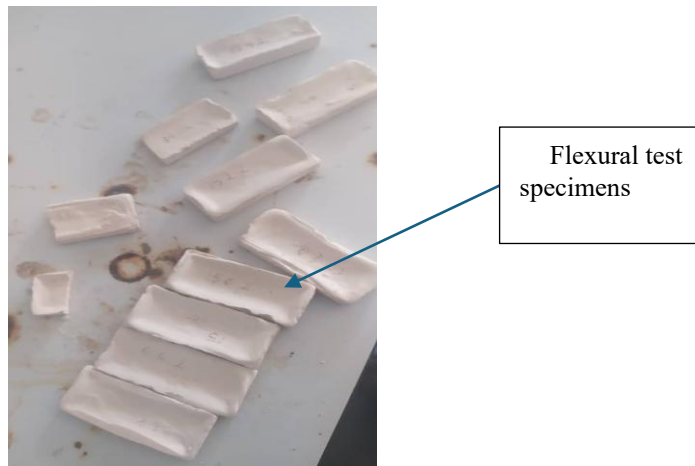


Fig. 3: Cooled Flexural Samples from the Oven

3. Results and Discussion

3.1 Flexural Strength

This is one of the parameters that are attributed to the performance of materials, a high flexural strength means the ceramic can withstand a considerable amount of tensile load up to its rupture (Dresch et al., 2021). Figure 4 highlights the results of the flexural analysis performed on sample B

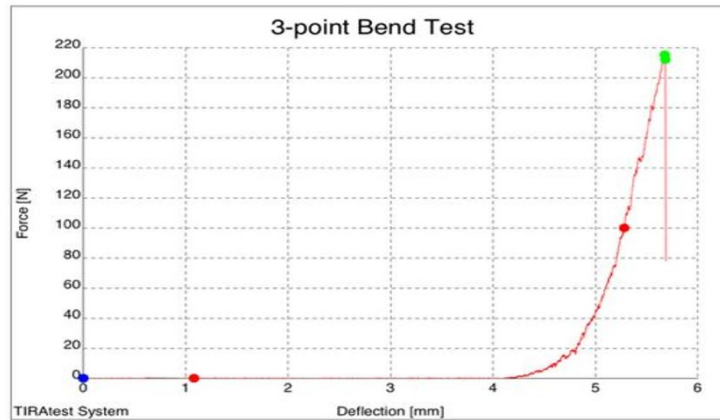


Fig. 4: Flexural Results of Sample B

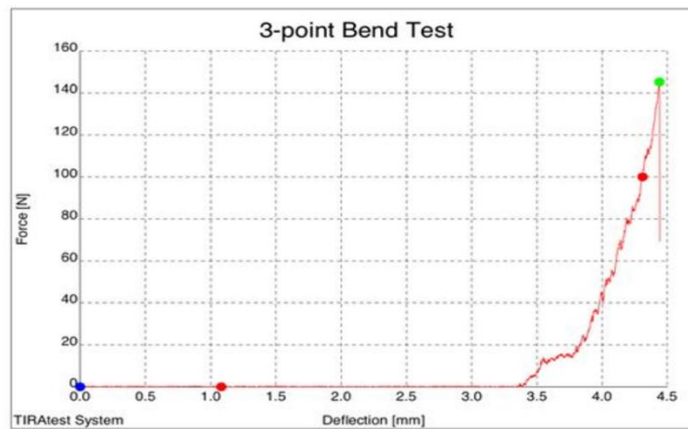


Fig. 5: Flexural Results for Sample C

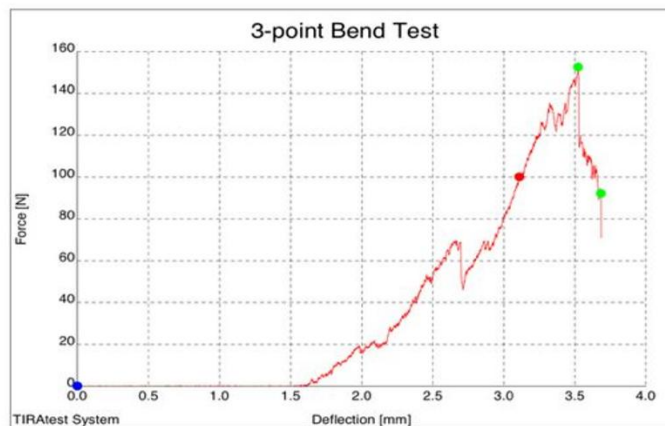


Fig. 6: Flexural results for Sample D

The flexural rigidity plots of the spinel samples are illustrated in Figures 5-6. Sample B exhibited the highest flexural strength at 210 MPa, while Sample D, shown in Figure 6, recorded 150 MPa, and Sample C, depicted in Figure 5, had a strength of 145 MPa. These results were similar to findings of (Blumer & Rittel, 2018). Sample A failed to reach the laboratory for testing due to unforeseen circumstances during transportation.

3.2 Characterization of the Samples

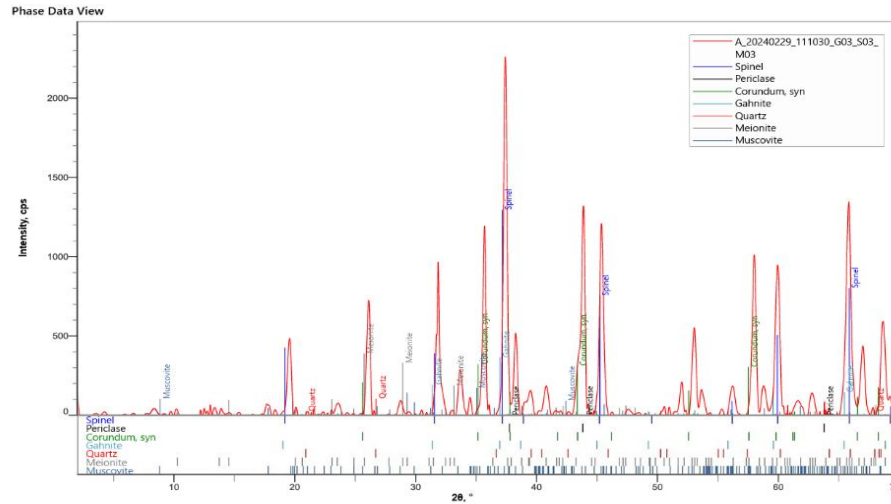


Fig. 7: XRD Result for Sample A

Figure 7 presents the XRD results for Sample A, illustrating the composition of spinel after sintering at 1600 °C. The analysis indicates that spinel constituted 36.9% of the composition, while periclase was found at 29.5%, and corundum at 24.7%. Additionally, trace elements such as garmite, quartz, meriosite, and muscovite exhibited a similar pattern (Ptáček et al., 2013; Al-Omari et al., 2024).

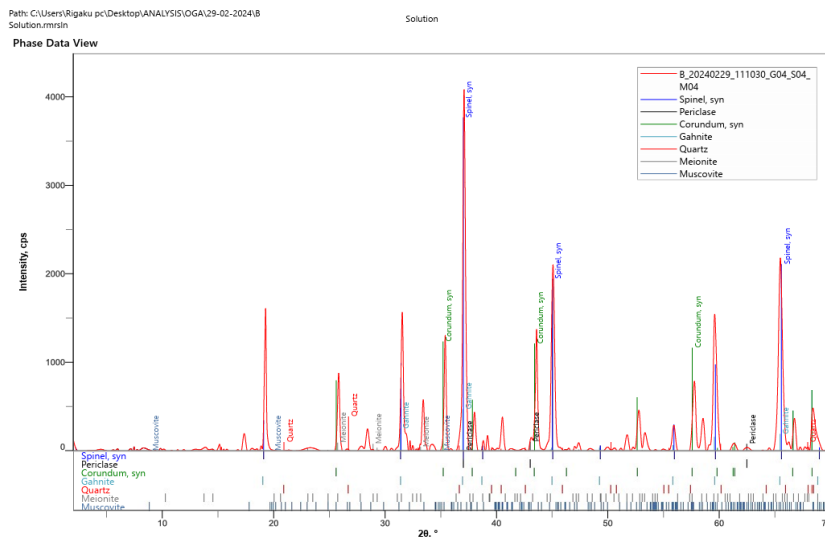


Fig. 8: XRD Results for Sample B Spinel

According to Figure 8, sample B exhibits the formation of spinel, comprising 52% spinel, 13.1% periclase, 27% corundum, along with various trace elements.

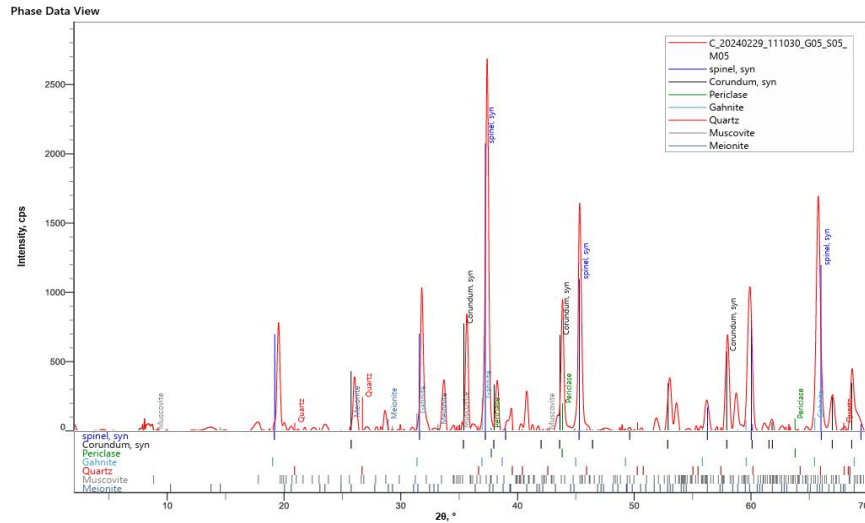


Fig. 9: XRD Results for Sample C

Figure 9 presents the XRD results for sample C, which consists of 58.9% spinel, 23.5% corundum, and 9.0% periclase, along with various trace elements.

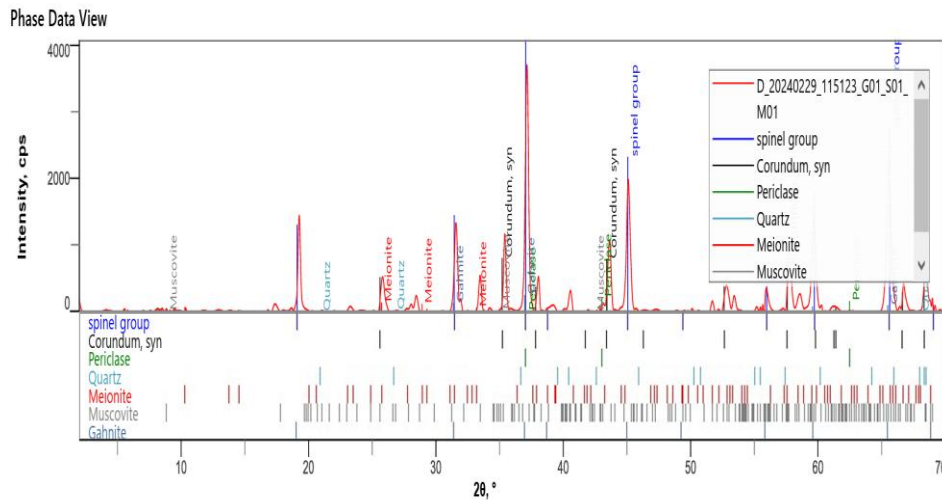


Fig. 10: XRD Result of Sample D

Figure 10 presents the results for sample D, indicating that 63% spinel was formed, along with 23.5% corundum, 9.0% periclase, and other trace elements. The X-ray diffractograms for samples A-D, shown in Figures 7-9, illustrate the crystallographic phases present in the spinel ceramics. The diffractograms of the samples sintered at 1600°C reveal a progressive formation of spinel, with percentages of 36.9%, 52%, 58.9%, and 63% respectively.

In Figure 7, sample A displayed the highest intensity peaks at 2-theta (2θ) values of 59.92° and 56.09°, while lower intensity peaks were noted at $2\theta = 45.34^\circ$, 37.42°, and 31.84°. Sample B, depicted in Figure 8, showed high-intensity peaks at $2\theta = 65.42^\circ$, 59.55°, and 55.86°, with lower intensity peaks at $2\theta = 45^\circ$, 37°, 35.37°, and 19.15°. Sample C, illustrated in Figure 9, had high-intensity peaks at $2\theta = 65.69^\circ$, 59.79°, and 56.13°, and lower intensity peaks at $2\theta = 45.26^\circ$, 37.37°, and 31.74°.

Sample D, shown in Figure 10, exhibited the highest intensity peaks at $2\theta = 65.56^\circ$, 59.64° , and 55.90° , while the lowest intensity peaks were at $2\theta = 45^\circ$, 37° , and 31° . Corundum ($\alpha\text{-Al}_2\text{O}_3$) was a prominent phase in all samples. In sample A, high-intensity peaks were observed at $2\theta = 57.97^\circ$ and 52.99° , with a low-intensity peak at $2\theta = 35.64^\circ$. Sample B showed high-intensity peaks at $2\theta = 57.69^\circ$ and 52.73° , while low-intensity peaks were recorded at $2\theta = 37.99^\circ$, 35.27° , and 25.78° . Sample C had high-intensity peaks at $2\theta = 68.57^\circ$, 66.90° , 57.93° , and 52.99° , with lower intensity peaks at $2\theta = 43.80^\circ$, 38.22° , 35.67° , and 26.03° . Finally, sample D recorded high-intensity peaks at $2\theta = 66.68^\circ$ and 57.72° , and low-intensity peaks at $2\theta = 43.60^\circ$, 35.34° , and 25.84° .

Periclase crystals were identified in samples A-D, but the trend in crystal formation varied among these samples. In sample A, periclase accounted for 29.5% of the formation and was detected at a low-intensity peak of $2\theta = 38.28^\circ$. Sample B showed a lower formation percentage of 13.17% at $2\theta = 43.57^\circ$. In contrast, samples C and D exhibited much lower formation rates of 9% and 2.87%, respectively, rendering them undetectable by X-ray analysis. This indicates that periclase formation is more prevalent in mixtures with a higher magnesia content compared to alumina. The interaction between magnesia and alumina leads to the development of the cubic structure typical of spinel-phase ($\text{Mg-Al}_2\text{O}_4$). (Sickafus et al., 1999; International Union of Crystallography, 1952; Seko et al., 2006; Hill et al., 1979). The percentage compositions presented in samples A-D align with the stoichiometric formulations of spinel (Adumbukulath et al., 2022; Blumer & Rittel, 2018; Ganesh, 2013; Ganesh et al., 2011).

3.3. Scanning Electron Microscope (SEM)

Figure 11 illustrates the SEM micrographs at a scale of 50 microns, revealing a denser structure and more compact pores compared to the earlier sample that contained different variations of magnesia.

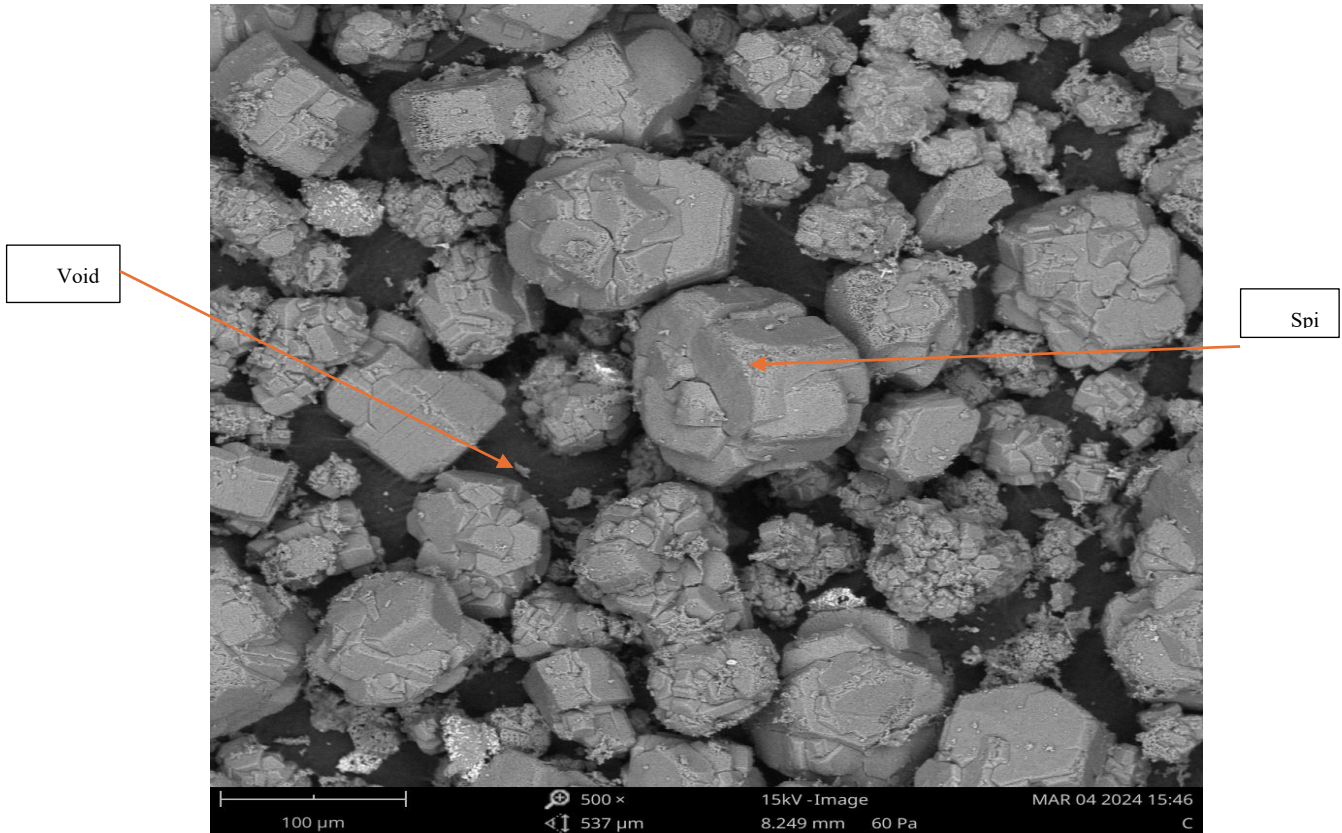


Fig. 11: SEM results for Spinel sample C at 100 microns

Figure 12 illustrates the scanning electron microscope (SEM) image at a magnification of 100 microns for sample C, which is composed of 20 grams of magnesia and 75 grams of alumina. The observed morphology resembles that of spinel phases; however, the particles appear more dispersed and exhibit incomplete densification, as indicated by the arrows pointing to the spinel particles and the void spaces.

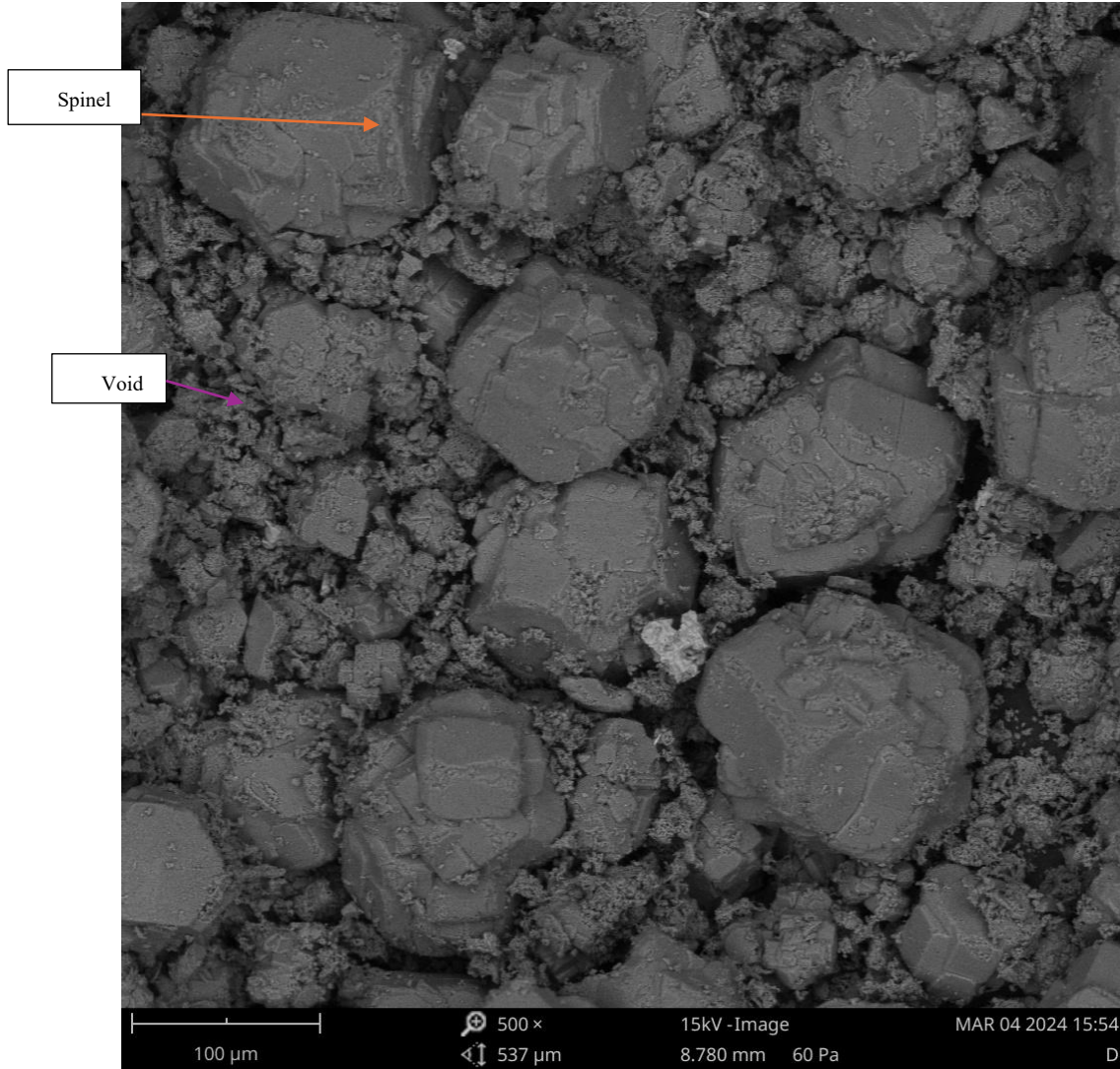


Fig. 12: SEM Results of Spinel Sample D at 100 microns

Sample D, consisting of 25 g of magnesia and 100 g of alumina, aligns with the typical spinel formulations, exhibiting more continuous pores and enhanced densification, as illustrated in Figure 12. In this figure, the orange arrow points to the spinel particles, while the blue arrow highlights the void spaces. As the temperature rises, the area of the MgAl_2O_4 phase expands, leading to a significant increase in the amount of alumina (Wang et al., 2015; Wang et al., 1992). This is demonstrated in the SEM results for sample D, which had a greater alumina composition and achieved a higher spinel formation percentage of 63%. It was also observed that sintering aids and higher magnesia content increase the densification of spinel, which is evident in the formulation of Sample D (Ganesh, 2013; Ganesh et al., 2011).

The method used to fabricate the spinel may contribute to the reduced densification and the presence of discontinuous pores in the samples' morphology, according to researchers (Adumbukulath et al., 2022;

Blumer & Rittel, 2018; Dericioglu & Kagawa, 2003; Browne et al., 2009; Macaigne et al., 2018) the studies reported similar findings; however, the morphologies noted in these studies exhibited greater continuity and complete densification, which can be attributed to the production methods used, including pressured sintering, sol-gel processes, 3D printing, hot isostatic pressing (HIP), and hot pressing (HP).

Spinel exhibits remarkable magnetic, optical, electrical, and catalytic properties due to its unique composition, electron configuration, and valence states. Various spinel compounds possess distinct characteristics; for example, those containing Fe, Co, Cr, and Ni primarily demonstrate magnetic behaviors such as ferrimagnetism and paramagnetism (Zhao et al., 2017; Zviagin et al., 2016; Sonoyama et al., 2006; Phanichphant, 2012). The possible uses of these spinel compounds include applications in information technology for data storage, magnetic sensors, and the electrical and electronics sectors, particularly in the creation of devices like supercapacitors and metal-ion batteries (Marco et al., 2001; Whittingham, 2004).

Recent studies have investigated spinel compounds as potential electrode materials for sodium-ion, magnesium-ion, and zinc-ion batteries (Sonoyama et al., 2006; Whittingham, 2004; Wei et al., 2016). Another benefit of spinel compounds is their effectiveness as catalysts in a range of reactions, including the oxygen reduction reaction (ORR), oxygen evolution reaction (OER), ammonia oxidation (NH₃), urea oxidation, and several others (Zhao et al., 2017; Molla et al., 2014). These catalytic processes utilizing spinel catalysts have demonstrated greater efficiency in energy conversion and storage systems, including fuel cells and metal-air batteries (Zhao et al., 2017; Butt & Rehman, 2019; Živcová-Vlčková et al., 2012). Transparent spinel exhibits optical characteristics such as electrochemical luminescence, photoluminescence, and unwanted light absorption, making it suitable for use in laser devices (Blumer & Rittel, 2018). Magnesium aluminate spinel (MAS) serves as a substitute for conventional chromate refractories in steel ladles and is also utilized as thermal barrier coatings for blades and vanes in gas turbines, as well as in cement rotary kilns (Ganesh, 2013).

The future is embracing sustainability by utilizing spinel compounds not only as decorative elements but also as crucial catalysts for achieving clean energy standards, potentially substituting noble metal catalysts either partially or entirely. Additionally, spinel can serve as electrodes in rechargeable batteries. As a result, spinel compounds offer a wide range of benefits and applications across various industries.

Table 2: Table of Comparison between obtained results and Available Literature

S/N	Flexural Strength (MPa)	Mode of production	Application	Source
1	155-330	Hot Isostatic Pressing and slip-casting	Infra(red) windows, opto-electric components, lamp envelopes, transparent armours.	(Zubrzycka et al., 2024)
2.	172-190	Co-doping	Military application	(Zan et al., 2023)
3.	150-300	Hi-pressure Spark Plasma sintering (HPSPS)	Enhance catalytic performance, doping with metals and non-metals, surface modification of spacecraft,	(Sokol et al., 2017)
4.	Our Samples 145-210MPa for Sample C, D, and B	Slip casting		Obtained Results

Table 2 presents a comparison of our results with the existing literature. Our findings highlight the potential of the studied materials for various advanced applications, as detailed in the table. The results from the analysed samples indicate that the spinel is well-suited for improved catalytic uses, military applications, lamp envelopes, and numerous other structural applications.

4. Conclusions

The development of spinel-based technologies is expected to improve operational capabilities and contribute to national security, economic diversification, and technological progress. This study acts as a foundational step for spinel research and development (R&D) in Nigeria, supporting the country's potential for local spinel manufacturing. The findings indicated that samples A, B, C, and D exhibited characteristics consistent with spinel formation, with sample D showing the highest formation rate at 68%, as reflected in its composition. The production of spinel was significantly influenced by impurities in alumina, which reduced both the strength and formation of spinel. Additionally, the chosen method of Slip Casting resulted in less-densified spinel due to its pressureless nature, and the alumina, derived from kaolin, had lost its plasticity, necessitating the use of titanium oxide as a sintering aid. The study recommends integrating a pressured sintering method with the slip casting technique to produce a more densified spinel, which would provide enhanced flexural strength for various applications.

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