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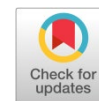
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Research article

Effect of different binders on microstructural evolution and strength of sol-gel bonded Al_2O_3 -spinel castables



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ABSTRACT

Al_2O_3 -spinel castable refractories offer several benefits, including high refractoriness, strong resistance to chemical attack, and excellent mechanical strength. These properties make them a preferred choice for steel ladle lining below the slag line. In this study, the effect of different binders on Al_2O_3 -spinel castable refractory was investigated. Three sol systems, including alumina, spinel, and silica sol, were separately employed as bonding agents in ultra-low cement castable formulations. Key properties, such as phase composition, microstructure, and mechanical performance of castable refractories, including bulk density (BD), apparent porosity (AP), and cold compressive strength (CCS), were evaluated. The properties of the castables with sol-gel bonding were compared with those with hydraulic bonding (calcium aluminate cement). It was observed that the castables containing silica sol resulted in higher cold compressive strength (2103 kg/cm^2) and higher bulk density because of the absence of low melting or eutectic phases and synthesis of the mullite phase.

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KEYWORDS

Alumina-spinel castable
Alumina sol
Spinel sol
Silica sol
Cement



1. Introduction

Refractories can withstand high temperatures, pressure, and chemical interactions without undergoing significant chemical or physical changes. They are mainly employed in the iron, steel, and petrochemical industries, playing a pivotal role in both product quality and production costs. Alumina-spinel (Al_2O_3 - MgAl_2O_3) refractories, a subclass of alumina-based materials, are widely employed for ladle lining applications. These refractories boast excellent refractoriness, resistance to chemical and thermomechanical stresses, non-wettability by molten steel and slag, high mechanical strength, low thermal expansion, and resistance to hydration. [1–2]. Due to these features, Al_2O_3 -spinel refractory materials are mostly used as steel ladle linings below the line of slag, refining furnaces and ladles, pocket block of purging plugs, high-temperature industries of metallurgy (both ferrous and nonferrous), cement, glass, etc. [3–6]. Among various unshaped refractories, the ordinary refractory castables were bonded with calcium aluminate cement (CAC) containing up to 20% CaO. The

Al_2O_3 , CaO, and SiO_2 reaction results in low melting temperature phases like gehlenite and anorthite. Consequently, the presence of CaO compromises the high-temperature performance of these refractories and restricts their applications in extreme environments [7–9]. Additional challenges in cement-based formulations arise during curing and dewatering processes, where poor management can result in cracking or explosive spalling. To circumvent these issues, efforts have focused on reducing cement content. Alternative binders such as hydratable alumina (HA), calcium aluminate (CA), monoaluminum phosphate, and sodium silicate have been explored to replace traditional cement-based systems [10–12].

The advancement of the sol-gel method has created a novel era in the bonding system for refractory castable technology. The basic of sol-gel bonding is creating a 3D network (gel) of particles surrounding the refractory particles. After heat treatment, this gel transforms into ceramic bonds and affects the properties of the refractory [13]. Various sol systems—including mullite, alumina, and silica sols—have been tested as alternative binders for low or no-cement refractory

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formulations [14–17]. Among these, silica sol has gained considerable commercial traction for monolithic and castable refractories due to its stability, high solid content, easy availability, and ability to promote mullite formation at lower temperatures. Dasgupta et al. [7] synthesized silica sols with different routes via a sol-gel system using inorganic, organic, and ion-exchange processes. They used silica sols as a binder in low-cement alumina castable. Their results revealed that the synthesized silica sol by the cation exchange process has a significantly good influence on the characteristics of low-cement alumina castable with 3% addition. The factors that favor its wide application are higher stability and its high solid amount, and the possibility of mullite synthesis at low temperatures. Despite the advantages, the presence of free silica makes them acidic. Thus, silica sol bonded castables were not recommended for basic environments like steel plant applications. Emerging research has increasingly shifted toward alternative solutions with improved high-temperature performance. It was reported that using alumina sol as a binder in refractory castables results in a highly pure refractory system that hinders the probability of any low-melting phase synthesis in the castable. So, it improves the properties and corrosion resistance of the refractory castables at high temperatures. Mukhopadhyay et al. [18] studied the application of cost-effective alumina sol as a no-cement binding agent in refractory castables prepared using a simple tapping method. Their findings underscore the potential for alumina sol to become a key player in advancing refractory material technologies while addressing critical performance limitations. They observed that the apparent porosity, bulk density, and cold-crushing strengths of sol-bonded castables at high temperatures are better than those with cement bonding. Also, they reported [19] a comparative investigation of silica and alumina sols as a binder in ultra-low cement castables and reported that excellent homogeneity and microstructural control are feasible with the alumina sol binder at high temperatures. Therefore, the strength of alumina sol-bonded castables is greater than the silica sol-bonded ones. Ghosh et al. [20] investigated the some types of inorganic sol and gel materials on high alumina-based low and ultra-low cement refractory castables. They observed that sol-gel bonded refractory castable renders excellent installation flexibility and uniform distribution of fine powders with the development of good phases at relatively low temperatures. Also, they reported that the sol-gel bonded refractory castable could show desirable spalling resistance.

The present research aims to investigate the performance of castables with varying sol-gel bonding agents—namely alumina, spinel, and silica sols—and compare their properties to those of castables bonded with hydraulic cement (calcium aluminate cement, CAC). Critical properties, such as phase composition, microstructure, and mechanical characteristics (bulk density, apparent porosity, and cold compressive strength), were systematically characterized.

2. Experimental

2.1. Starting materials and methods

In the present study, to prepare the ultra-low cement castable (ULCC) Al_2O_3 -spinel refractories, alumina sol, silica sol, spinel sol, and high alumina cement were used as binders. The spinel and alumina sols were synthesized through an inorganic methodology, whereas the silica sol was a commercially sourced product. The specific properties of these sols are listed in Table 1 [14–15].

Table 1. Properties of alumina, spinel, and silica sols applied as binder.

Sol	Alumina sol	Spinel sol	Silica sol
Appearance	Translucent	Translucent	Translucent
pH	3–4	3–4	8–9
Solid content (%)	6	6.5	12.8
Density (g/cm^3)	1.19	1.29	1.13
Average particle size at 1000 °C (nm)	30–40	~ 45	50
Phase analysis at 1000 °C	α -alumina	Spinel	Cristobalite

Four different Al_2O_3 -spinel castable refractories were formulated using raw materials such as white tabular alumina (with varying sizes of particle, purity of 98%, Almatix), calcined alumina (purity of 98%, NFC, China), fused spinel (0–0.5 mm, <0.075 mm, 20% MgO), reactive alumina (purity of 98%, 20 μm , Almatix), microsilica (microsilica Grade 95, China), calcium aluminate cement (2.8 μm , 80 mass% Al_2O_3 and 17.2 mass% CaO, Denka Co.), and binders such as alumina sol [15], spinel sol [14], silica sol (Barsam Shimi, Iran). Hexametaphosphate was used as a dispersing agent. The particle-size distribution of the raw materials was optimized using Andreasen's packing model; details are provided in Table 2. For castables bonded with sols, the binder content was added over the baseline 100%. Additionally, in sol-bonded samples, a 1% surplus of cement was included to enhance setting.

The preparation process began by thoroughly mixing all raw materials except for the sol and water in a Hobart (10 Quart C100 Mixer) for 5–6 minutes to achieve uniformity. Then, the sol and water were added to the batch and mixed for 5 min, and the "ball-in-hand" route was applied to check the workability of the castable. Samples were then shaped into $5 \times 5 \times 5 \text{ cm}^3$ cubes through vibration casting. The content of required water varied with the binder. After casting, samples containing 7% cement were cured under humid conditions for 24 h at room temperature and then air-dried for another 24 h. Then, all of the castables were dried in an oven (Tebzaman) at 110 °C for 24 h. Finally, the castable specimens were fired at different temperatures (500, 1000, 1400, and 1650 °C) with a 7 °C/min heating rate for 3 h soaking time at the peak temperatures. The flow diagram for the steps of preparing Al_2O_3 -spinel castable refractory is given in Fig. 1.

2.2. Characterization

The dried and fired specimens underwent various evaluations, including apparent porosity (AP), bulk density (BD), cold compressive strength (CCS), phase study via X-ray diffraction (XRD), and microstructure investigation by scanning electron microscopy (SEM). The bulk density and apparent porosity of the castable specimens were estimated by Archimedes' technique in the water. The CCS of dried and fired castables was conducted by a 60-N universal Zwick Roell machine with a loading rate of 25 kN/min. XRD analysis was done on the matrix components of specimens fired at 1650 °C using a D5000 Siemens X-ray diffractometer outfitted with a copper anode (Cu-K α radiation; $\lambda=0.15405 \text{ nm}$) operating at 40 kV/30 mA and scanning

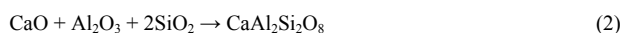
Table 2. Batch composition of Al₂O₃-spinel refractory castables with different binders.

Batch materials		Alumina sol	Spinel sol	Silica sol	Cement
Tabular alumina	(3–5 mm)	26	26	26	26
	(1–3 mm)	17	17	17	17
	(0.5–1 mm)	11	11	11	11
	(0–0.5 mm)	6	6	6	6
	<0.075 mm)	10	10	10	3
Spinel	(0–0.5 mm)	11	11	11	11
	(<0.075 mm)	4	4	4	4
Reactive alumina		5	5	5	5
Calcined alumina		5	5	5	5
Microsilica		5	5	5	5
Calcium aluminate cement		1	1	1	7
Sol		7	7	7	0
Water		4.5	6	0	5
Dispersant agent		0.1	0.1	0.1	0.1

at a rate of 2 °/min within the range of 20=20 °–80 °. Microstructural observations and energy-dispersive spectrometry (EDS) were carried out using a field-emission SEM (FESEM Mira3 Tescan) equipped with a DXP-X10P X-ray processor. The microstructure and chemical compositions of the specimens were determined using field emission scanning electron microscopy (FESEM, Mira3 Tescan, Czech Republic) and energy-dispersive spectrometry (EDS, Digital X-Ray Processor, DXP-X10P).

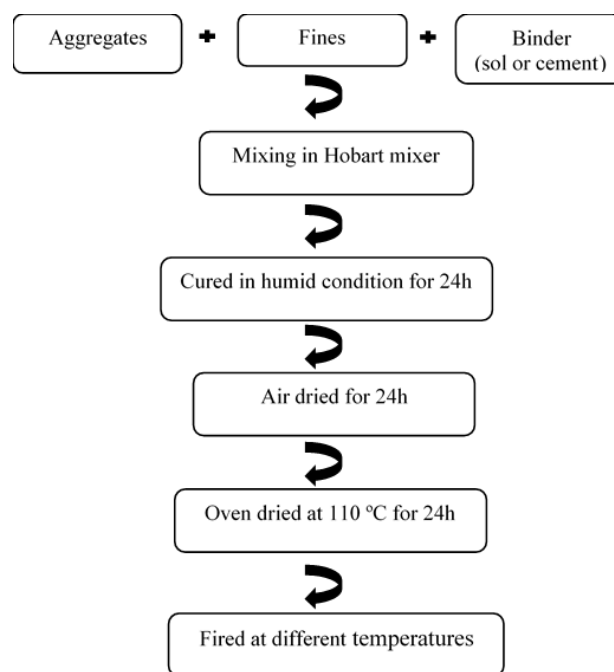
3. Results and discussion

To study the phase composition of the castable refractories, XRD diffraction patterns of the matrix of the castables were prepared. Figs. 2–5 illustrate XRD results for samples containing various binders fired at 1650 °C. Fig. 2 illustrates the X-ray diffraction (XRD) patterns of a refractory castable matrix containing cement fired at 1650 °C. The primary phases detected were Al₂O₃ and MgAl₂O₄. In addition, trace amounts of mullite, calcium hexa-aluminate (CA₆, CaAl₁₂O₁₉), and anorthite (CaAl₂Si₂O₈, 1550 °C) were observed. Mullite and anorthite phases synthesized as a result of the reactions between microsilica and free CaO with Al₂O₃, as described by reactions 1 and 2 [21].



Calcium aluminate cement reacted with alumina to produce calcium hexa-aluminate (CA₆) in the cement-containing refractory castable matrix at temperatures above 1350 °C [22]. The formation of CA₆ causes significant modifications in the microstructure and properties of the castables upon firing. Based on the XRD patterns of the castables matrix containing silica, alumina, and spinel sol, which fired at 1650 °C (Figs. 3, 4, and 5), the main phases of alumina (Al₂O₃) and spinel (MgAl₂O₃) were detected. Mullite appeared as a secondary phase

in these patterns due to the reaction of microsilica with Al₂O₃ in the matrix. However, in the XRD pattern of castable matrices bonded with silica sol, minor peaks of free silica were also observed.

**Fig. 1.** Flow diagram for the steps of preparing Al₂O₃-spinel castable refractory.

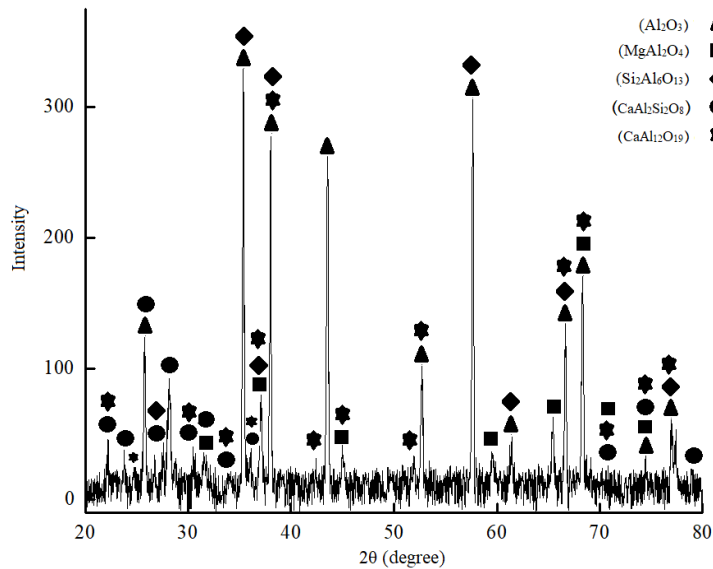


Fig. 2. XRD analysis of the matrix part of fired Al_2O_3 - MgAl_2O_3 castables at 1650 °C containing cement.

Fig. 6 exhibits the changes in the CCS values of refractory castables with different binders across various firing temperatures. Dried castables at the temperature of 110 °C own a very low green strength. Cement-bonded refractory castables had the highest strength due to robust hydraulic bonding. Refractory castables with sol-based bonding showed lower strength in comparison, likely because of weak coagulation bonding inherent to sol-gel systems. The strength in sol-gel bonding arises primarily from the coagulation of colloidal particles surrounding refractory materials and from the solid content within the sol. According to Table 1, silica sol has a solid amount of 12.8%,

which is remarkably higher than the other sols, resulting in higher green strength and more effective coagulation bonding. Conversely, refractory castables bonded with alumina or spinel sols exhibited lower strength for two key reasons: their lower solid content (resulting in weaker coagulation bonding) and higher water demands required to achieve comparable flow consistency. Increased water content leads to higher porosity, reducing the strength of the castables. Within the temperature extend of 400–1000 °C, sol-gel-bonded refractory castables demonstrated increasing strength due to nanoparticle crystallization within the sols. The sol-gel bonding mechanism involves

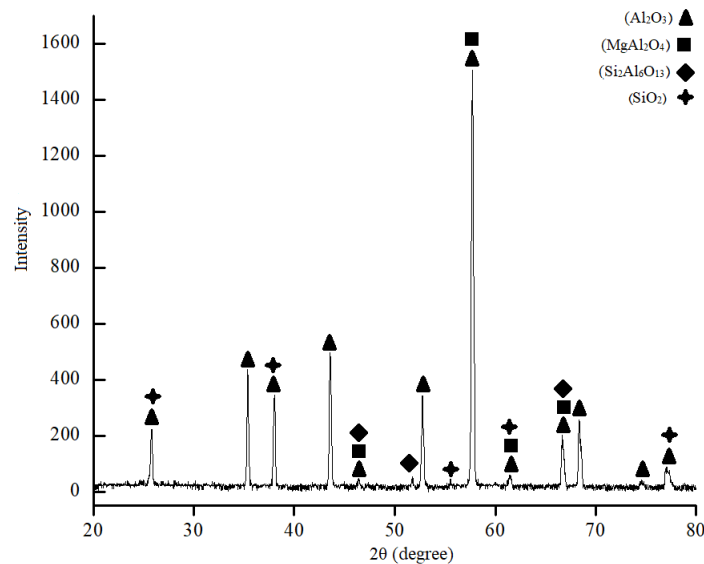


Fig. 3. XRD analysis of the matrix part of fired Al_2O_3 - MgAl_2O_3 castables at 1650 °C containing silica sol.

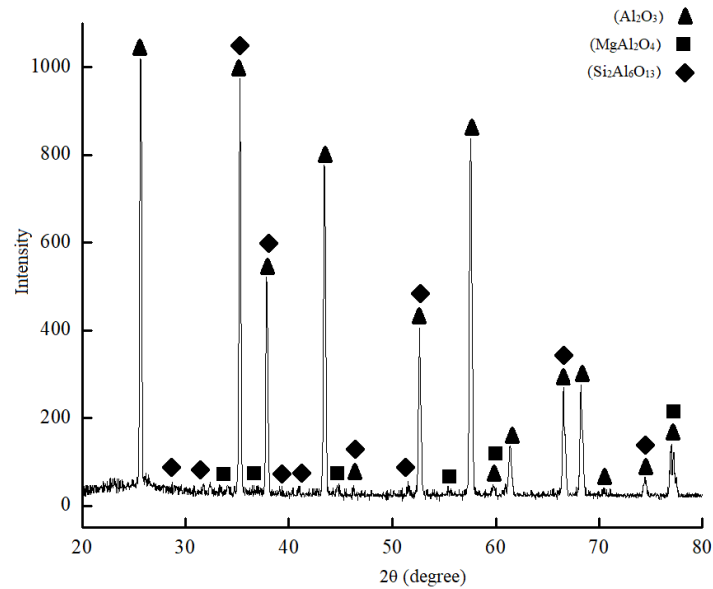


Fig. 4. XRD analysis of the matrix part of fired Al_2O_3 - MgAl_2O_3 castables at 1650 °C containing alumina sol.

forming a three-dimensional gel network that envelops refractory materials and contributes to strength development during drying. Subsequent heating of the refractory castable develops ceramic bonding due to sintering at low temperatures, resulting in superior properties than cement-bonded refractory castable. Also, the reaction between very fine (nanometer size) particles in the sol and fine particles in the refractory castables may start the sintering process in the refractory castables, which increases the strength at 1000 °C and produces mullite phases. At this temperature, CCS values for silica-sol-

bonded and cement-bonded refractory castables were measured at 1663.66 kg/cm² and 1625.33 kg/cm², respectively.

At temperatures exceeding 1400 °C, the silica sol bonded refractory castable has cold compressive strength higher than the cement bonded refractory castable. This is because cement-bonded systems undergo a reaction between cement and fine alumina particles in the matrix to form CA_6 , causing a theoretical volumetric expansion of +3.01%. If unmanaged, this volumetric change can induce cracking within the material system. By contrast, colloidal binders exhibit greater

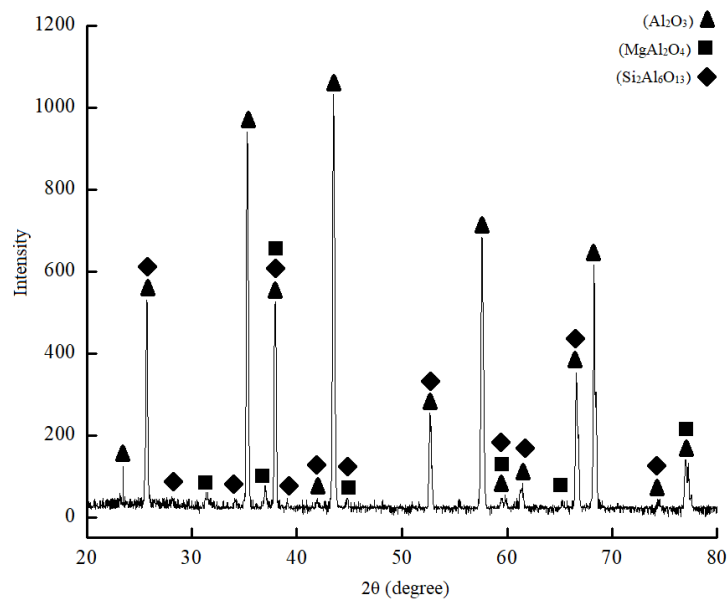


Fig. 5. XRD analysis of the matrix part of fired Al_2O_3 - MgAl_2O_3 castables at 1650 °C containing spinel sol.

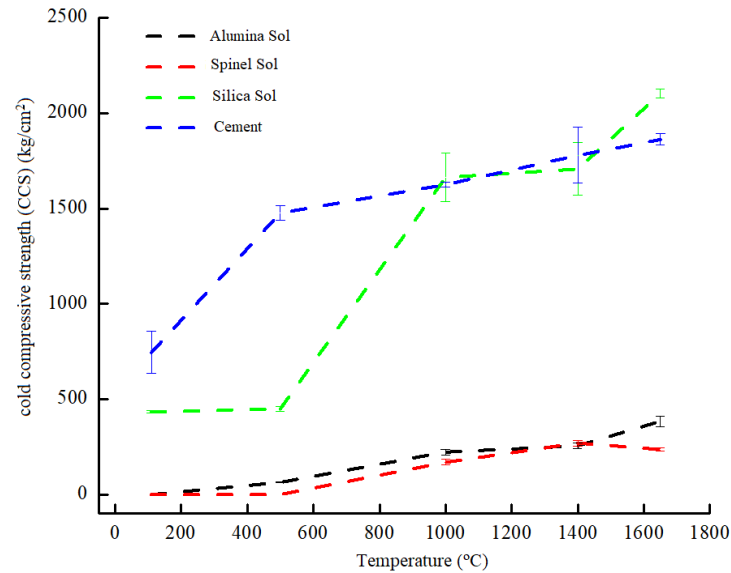


Fig. 6. Cold compressive strength (CCS) of the Al_2O_3 - MgAl_2O_3 castables containing different binders fired at different temperatures.

volumetric stability upon firing. Additionally, in cement-bonded refractories, the formation of low-melting-point phases, such as anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$), as noted in Fig. 2 phase analysis, contributes to reduced strength values.

The bulk density variations of refractory castables with various binders, dried and fired at varying temperatures, are illustrated in Fig. 7. A usual trend of increasing bulk density values with rising temperatures is observed across the binders. This increase is attributed to enhanced bonding between ultrafine sol powders and fine matrix components. Fig. 7 shows that the bulk density of alumina and spinel sol bonded

refractory castables at the green stage (110 °C) cannot be measured due to very low green strength. Among the refractory castables, silica sol-bonded ones exhibit the highest bulk density. This is due to the rapid gelling effect caused by the high reactivity of colloidal silica particles, which form a homogeneous atomic-level mixture, as well as the higher solid content and lack of water requirement for similar flowability. At intermediate temperatures (~1000 °C), sol-bonded refractory castables show faster increases in bulk density compared to cement-bonded ones, owing to the breakdown of cement hydrate bonds. At higher temperatures (>1400 °C), silica sol and cement-bonded refractory

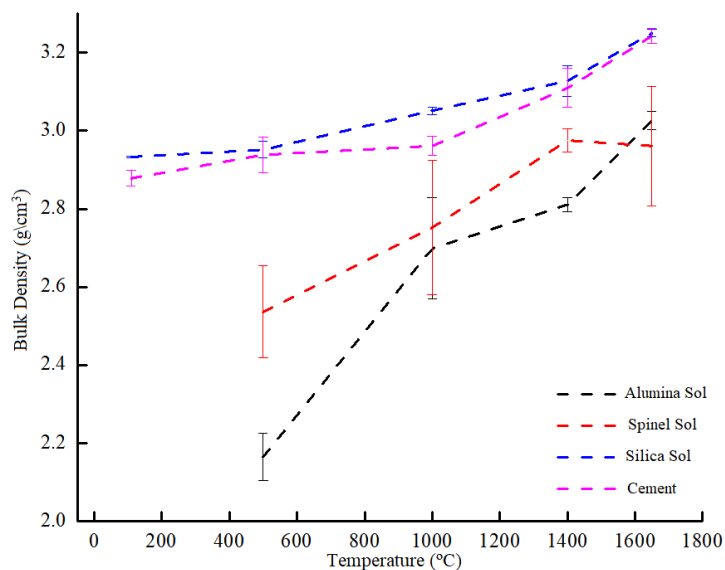


Fig. 7. Bulk density of the Al_2O_3 - MgAl_2O_3 refractory castable containing different binders at different temperatures.

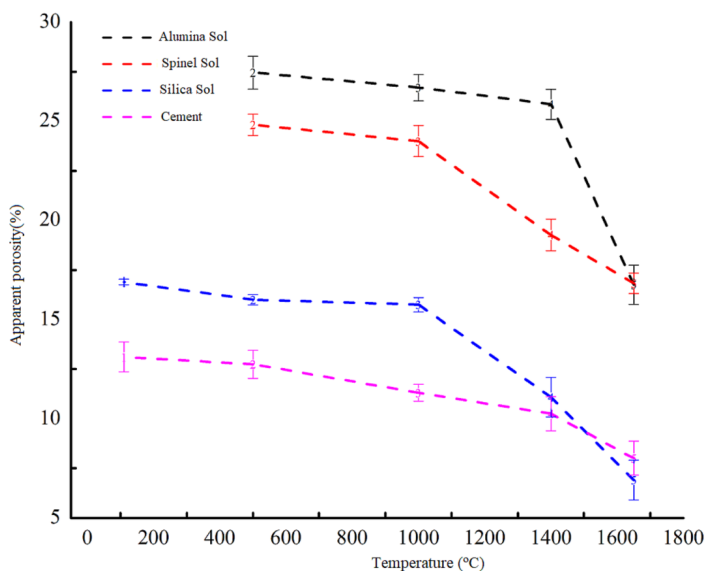


Fig. 8. Apparent porosity of the Al_2O_3 - MgAl_2O_3 refractory castable containing different binders at different firing temperatures.

castables achieve the highest bulk densities, with readings around 3.2 – 3.3 g/cm^3 at $1650 \text{ }^\circ\text{C}$.

Fig. 8 presents the apparent porosity (AP) values of refractory castables with various binders at various firing temperatures. The trend in apparent porosity is inverse to that of bulk density; it decreases with increasing temperature due to the progressive formation of ceramic bonds. Apparent porosity reaches its lowest values at $1650 \text{ }^\circ\text{C}$ for all castables. However, alumina and spinel sol-bonded castables exhibit higher apparent porosity compared to silica sol and cement-bonded alternatives, attributed to their lower solid content and higher water addition requirements. At $1650 \text{ }^\circ\text{C}$, the apparent porosity values are approximately 16%, 16%, 7%, and 8% for alumina, spinel, silica sols, and cement-bonded castables, respectively.

To analyze the microstructure, the fractured surfaces of fired refractory

castables were studied. Figs. 9 and 10 show the SEM micrographs of different bonded refractory castables fired at $1650 \text{ }^\circ\text{C}$. These images reveal a compact microstructure resulting from improved sintering between the refractory aggregates and the matrix phase. The enhanced grain-to-grain connection indicates significant densification because of the presence of finer sol powders. The element analysis of the cement-bonded refractory castable supports the presence of aluminum as the main element with some amount of Si, Ca, and O, showing the synthesis of calcium aluminate and mullite phases. There are calcium hexaluminate ($\text{CA}_6=\text{CaAl}_2\text{O}_{19}$) and mullite crystals synthesized locally as a result of the reaction between the CaO and SiO_2 and reactive fine Al_2O_3 from the castable matrix, as observed in the phase analysis study.

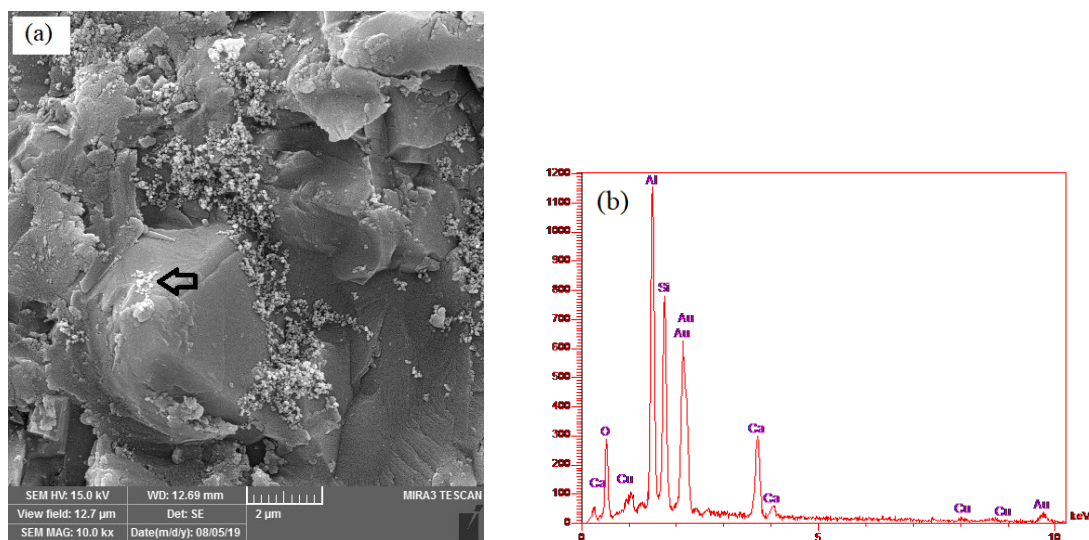


Fig. 9. SEM micrograph and EDS of the cement bonded refractory castable fired at $1650 \text{ }^\circ\text{C}$.

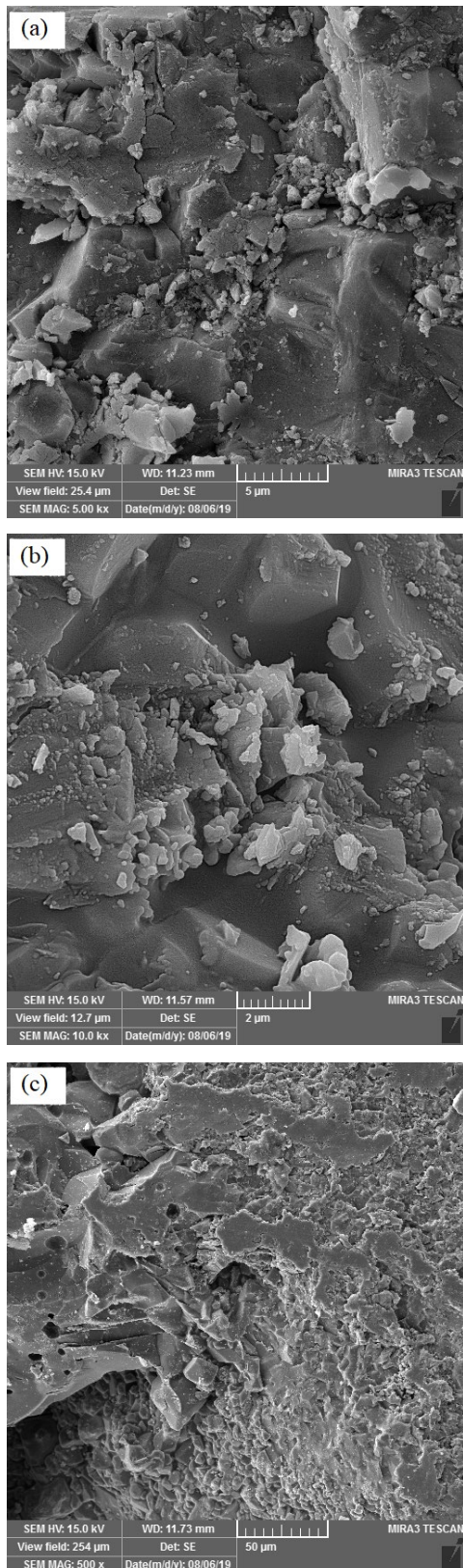


Fig. 10. SEM micrograph of refractory castable fired at 1650 °C: a) silica sol, b) alumina sol, and c) spinel sol bonded.

4. Conclusions

The study further explores the impact of different binders (spinel, alumina, and silica sols) on alumina-spinel refractory castables by comparing them to cement-bonded castables. The bulk density and apparent porosity of sol-bonded refractory castable at the green stage are lower than the cement-bonded refractory castable due to weak coagulation bonding and lower solid content of the sol. The densities and strength achieved in the sol-bonded refractory castable are impacted by the solid amount of the sols and the gelling tendency of the colloidal particles present in the sol. At elevated temperatures (1650 °C), silica sol-bonded castables demonstrate superior bulk density (3.2–3.3 g/cm³) and cold compressive strength (1663.66 kg/cm²) compared to alumina and spinel sol-bonded alternatives. These findings highlight a well-compacted and sintered microstructure for silica sol-bonded castables. The XRD pattern confirms the absence of low-melting phases in its microstructure. In cement-bonded refractory castable, due to the synthesis of low melting phases, especially anorthite (CaAl₂Si₂O₈), the values of strength are less than silica sol-bonded castables.

CRedit authorship contribution statement

Sahar Sajjadi Milani: Writing – original draft, Visualization, Investigation.

Mahdi Ghassemi Kakroudi: Writing – review & editing, Project administration.

Data availability

The data underlying this article will be shared on reasonable request to the corresponding author.

Declaration of competing interest

The authors declare no competing interests.

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