

The Effect of Fly Ash/Ground Granulated Blast Furnace Slag Ratio and Supplementary Microwave Curing on the Mechanical and Microstructural Properties of Alkali-Activated Materials

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Uçucu Kül/Öğütülmüş Granüle Yüksek Fırın Cürufu Oranının ve İlave Mikrodalga Kürünün Alkali Aktive Malzemelerin Mekanik ve Mikroyapısal Özellikleri Üzerindeki Etkisi

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Abstract

The curing regime plays a role in determining the performance and microstructural development of alkali-activated materials (AAMs). This study investigates the influence of supplementary microwave treatment following initial oven curing on AAMs prepared using fly ash (FA) and ground granulated blast furnace slag (GGBFS) in varying proportions. Three mixtures (100% FA, 50% FA-50% GGBFS, and 100% GGBFS) were produced using a constant $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratio of 1.0 and characterized by compressive strength, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), and mass loss measurements. Increasing the GGBFS content promoted the formation of amorphous reaction products, while supplementary microwave treatment had little effect on crystalline phases. FTIR indicated changes in the aluminosilicate bonding environment after alkali activation, whereas similar chemical functionalities were observed for the oven-cured control and microwave-treated specimens. GGBFS-containing specimens formed a denser reaction matrix but developed more pronounced microcracks, particularly following supplementary microwave treatment, whereas FA-rich mixtures contained numerous unreacted spherical particles. SEM-EDS indicated an increase in the Ca/Si ratio with increasing GGBFS content, reflecting greater calcium incorporation into the reaction products. The highest 28-day compressive strength was obtained for the 50% FA-50% GGBFS mixture, reaching 14.2 MPa for the oven-cured control and 11.2 MPa after microwave treatment. Supplementary microwave treatment resulted in lower 28-day compressive strength across all precursor compositions and was associated with greater mass loss and more pronounced microcrack formation. Overall, precursor composition exerted a greater influence on the investigated properties than supplementary microwave treatment, with the 50% FA-50% GGBFS blend providing the best balance of microstructural characteristics and mechanical performance.

Keywords: Alkali-activated materials; Ground granulated blast furnace slag; Compressive strength; Fly ash; Microwave curing

Öz

Kürleme yöntemi, alkali aktive malzemelerin (AAM) performansı ve mikroyapısal gelişimi üzerinde önemli bir etkiye sahiptir. Bu çalışmada, farklı oranlarda uçucu kül (FA) ve öğütülmüş granüle yüksek fırın cürufu (GGBFS) içeren AAM'lerde başlangıç geleneksel etüv kürünün ve etüv kürünü takiben uygulanan mikrodalga kürünün etkisi araştırılmıştır. %100 FA, %50 FA-%50 GGBFS ve %100 GGBFS olmak üzere üç karışım, sabit $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar oranı (1.0) kullanılarak hazırlanmış ve basınç dayanımı, X-ışını kırınımı (XRD), Fourier dönüşümlü kızılötesi spektroskopisi (FTIR), enerji dağılımlı X-ışını spektroskopisi ile taramalı elektron mikroskobu (SEM-EDS) ve kütle kaybı analizleri ile karakterize edilmiştir. GGBFS oranının artması ağırlıklı olarak amorf reaksiyon ürünlerinin oluşumunu teşvik ederken, kürleme yönteminin kristalin fazlar üzerinde belirgin bir etkisi gözlenmemiştir. FTIR analizleri alkali aktivasyon sonrasında alüminosilikat bağ yapısındaki değişimleri göstermiştir. GGBFS içeren numuneler daha yoğun bir reaksiyon matrisi oluşturmalarına rağmen, özellikle mikrodalga küründe daha belirgin mikroçatlaklar geliştirmiştir. SEM-EDS sonuçları, GGBFS oranındaki artışla birlikte Ca/Si oranının yükseldiğini göstermiştir. En yüksek 28 günlük basınç dayanımı, etüv kürü kontrol numunelerinde 14.2 MPa ve mikrodalga işlemi uygulanan numunelerde 11.2 MPa değerlerine ulaşan %50 FA-%50 GGBFS karışımında elde edilmiştir. Ek mikrodalga işlemi, tüm öncül bileşimlerde daha düşük 28 günlük basınç dayanımı ile sonuçlanmış ve bu durum, daha yüksek kütle kaybı ve daha belirgin mikroçatlak oluşumuyla ilişkilendirilmiştir. Sonuç olarak, öncül malzeme bileşiminin incelenen özellikler üzerinde ek mikrodalga işleminden daha belirleyici olduğu ve %50 FA-%50 GGBFS karışımının mikroyapısal özellikler ile mekanik performans açısından en dengeli sonuçları sağladığı belirlenmiştir.

Anahtar Kelimeler: Alkali aktive malzemeler; Yüksek fırın cürufu; Basma mukavemeti; Uçucu kül; Mikrodalga kürleme

1. Introduction

The construction industry is one of the largest contributors to global greenhouse gas emissions, with ordinary Portland cement (OPC) production accounting for approximately 7-8% of worldwide anthropogenic CO₂ emissions (Amran et al., 2020; Kunasegeran et al., 2025). The calcination of limestone together with the high energy demand required during clinker production has intensified the search for environmentally sustainable cementitious materials. Consequently, the development of low-carbon binders has become an important research priority in both academia and industry (Gartner and Hirao 2015; Pavlin et al., 2021).

Among the proposed alternatives, alkali-activated materials (AAMs), particularly those produced from industrial by-products such as ground granulated blast furnace slag (GGBFS) and fly ash (FA), have attracted considerable attention. These aluminosilicate-rich materials can be activated using alkaline solutions to form three-dimensional binding gels with excellent engineering performance while simultaneously reducing industrial waste disposal and greenhouse gas emissions (Deb et al., 2015; Ismail et al., 2014; Kurtulus and Kurtulus 2023; Kurtulus 2021). Compared with conventional Portland cement, AAMs generally exhibit lower embodied carbon, high compressive strength, low permeability, excellent chemical resistance, superior resistance to sulfates and chlorides, and enhanced durability under aggressive environmental conditions (Hardjito et al., 2004; Li et al., 2013). Thereby, their ability to incorporate industrial wastes also supports circular economy strategies by converting by-products into high-value construction materials.

The reaction mechanism of alkali activation involves the dissolution of aluminosilicate precursors, followed by the polycondensation of dissolved species into continuous binding gels. The dominant reaction products depend largely on the precursor composition. Slag-rich systems primarily form calcium-aluminosilicate-hydrate (C-(A)-S-H) gels, whereas fly ash-rich mixtures generally produce sodium-aluminosilicate-hydrate (N-A-S-H) gels (Liang et al., 2024; Pawluczuk et al., 2021; Wang et al., 2024). In blended GGBFS-FA systems, the synergistic interaction between slag and fly ash has been published to enhance microstructural development and mechanical performance compared with single-precursor systems. Therefore, blending GGBFS and FA has become an effective strategy for controlling the engineering properties of AAMs while promoting the sustainable utilization of industrial by-products (Ibrahim and Maslehuddin 2021; Mohamed 2023).

Recent studies have focused not only on improving the engineering performance of AAMs but also on optimizing their mixture design and predicting their mechanical behavior. Owing to the complex interactions among precursor chemistry, alkaline activators, liquid-to-solid ratio, and curing conditions, almost no universal mix design methodology has yet been established for AAMs. That means, practical guidelines for mixture proportioning continue to be developed to improve material performance and facilitate their large-scale implementation (C. Kurtulus and Baspinar 2022). Furthermore, predictive approaches have recently attracted considerable attention as efficient tools for estimating the mechanical performance of AAMs without relying exclusively on destructive compressive strength testing. For example, the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model has been successfully applied to predict the strength development of geopolymer materials, demonstrating its potential to reduce experimental effort and improve material design (Blázquez et al., 2018; Kurtulus and Baspinar 2022; Siyal et al., 2016). These studies collectively indicate that, although substantial advances have been made in mixture optimization and performance prediction, further research is needed to optimize curing strategies, particularly rapid-curing techniques such as microwave irradiation, to enable the practical, energy-efficient production of AAMs.

Nevertheless, curing remains one of the least-optimized processing parameters that affect the final performance of AAMs. Unlike Portland cement, many fly ash-based systems require elevated-temperature curing to achieve sufficient dissolution of aluminosilicate species and rapid geopolymerization. Conventional thermal curing is generally conducted at temperatures between 40 and 90 °C for 24 to 72 h, depending on precursor characteristics, activator composition, and mixture design (Davidovits 2011; Provis and Deventer 2014). Such prolonged curing durations increase energy consumption, reduce production efficiency, and limit the large-scale industrial application of AAMs.

In recent years, microwave curing has emerged as a promising alternative to conventional oven curing. Unlike conductive or convective heating, microwave irradiation generates volumetric heating through direct interaction between electromagnetic waves and polar molecules within the material (Atalay et al., 2024; Dong et al., 2024; Kastiukas et al., 2020; Nadeem et al., 2020). This mechanism enables rapid and relatively uniform internal heating, significantly reducing temperature gradients and shortening curing time. Microwave energy is converted

directly into thermal energy throughout the specimen, accelerating moisture migration, aluminosilicate dissolution, gel formation, and polycondensation reactions.

Several researchers have demonstrated the beneficial effects of microwave curing on AAMs. Aschoff et al., found that metakaolin-based geopolymers achieved mechanical properties comparable to conventionally cured specimens within only a few minutes of microwave exposure (Aschoff et al., 2024). Similarly, Watanabe and Kobayashi observed that optimized microwave curing promoted matrix densification, reduced pore volume, and improved mechanical performance (Watanabe et al., 2023). Other studies have further indicated that microwave curing may decrease drying shrinkage, refine pore structure, and improve early-age strength development while substantially reducing energy consumption (Dong et al., 2024). Nevertheless, excessive microwave power or prolonged exposure may induce rapid moisture evaporation, internal vapor pressure, microcracking, and strength loss, indicating that microwave curing parameters must be carefully optimized for each material system (Aschoff et al., 2024; Atalay et al., 2024; Dong et al., 2024).

Although microwave curing has been investigated for several alkali-activated systems, most available studies have focused on single precursors such as fly ash or metakaolin. Studies examining the effect of supplementary microwave treatment following initial thermal curing on blended FA-GGBFS systems with systematically varied precursor compositions remain limited (Aschoff et al., 2024). The novelty of this study lies in the systematic evaluation of supplementary microwave treatment across three FA/GGBFS precursor compositions, while maintaining a constant $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratio, thereby enabling assessment of the effects of precursor composition and subsequent microwave treatment on the mechanical and microstructural characteristics of AAMs.

In this study, AAMs with varying ratios of ground granulated blast-furnace slag and fly ash were initially subjected to identical oven-curing conditions. A portion of the specimens was subsequently exposed to supplementary microwave treatment, while the remaining specimens served as oven-cured controls. The effects of supplementary microwave treatment on the 28-day compressive strength, mass loss, phase characteristics, chemical bonding environment, and microstructural properties were systematically evaluated. The study aims to clarify how supplementary microwave

treatment influences FA/GGBFS-based AAMs with different precursor compositions and to assess its effects on their subsequent mechanical and microstructural development.

2. Materials and Methods

Ground granulated blast furnace slag (GGBFS) and Class F fly ash (FA) were used as aluminosilicate precursors for the production of AAMs. According to their chemical compositions, FA mainly consisted of SiO_2 (50.30 wt. %), Al_2O_3 (19.10 wt. %), and CaO (4.55 wt. %), whereas GGBFS contained 35.46 wt. % SiO_2 , 10.78 wt. % Al_2O_3 , and a considerably higher CaO content (40.57 wt. %). The markedly higher calcium content of GGBFS is expected to influence the reaction mechanism and the composition of the alkali-activated binding phases. The average particle sizes of FA and GGBFS were approximately 29.97 μm and 47.33 μm , respectively. The specific surface areas, determined using the Brunauer-Emmett-Teller (BET) method, were 7.91 m^2/g for FA and 0.93 m^2/g for GGBFS, while their specific gravities were 2.58 g/cm^3 and 2.88 g/cm^3 , respectively. The alkaline activator was prepared by mixing a commercial sodium silicate solution with sodium hydroxide (NaOH) in approximately 87 wt. % and 13 wt. %, respectively. Three different AAMs compositions containing varying proportions of GGBFS and FA were designed. To enable a direct comparison among precursor mixtures, the $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratio was fixed at 1.0 for all formulations by adjusting the amount of alkaline activator to match the chemical composition of each mixture. A constant $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratio was selected to minimize variations associated with activator composition and to enable a direct comparison of precursor composition and supplementary microwave treatment. Additional water was incorporated only when necessary to achieve adequate workability and homogeneous mixing without altering the targeted activator composition. The detailed mixture proportions are presented in Table 1.

Table 1. Mix design of alkali-activated binders, including precursor proportions, additional water, and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratio.

GGBFS (%)	FA (%)	Extra water (%)*	$\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$
50	50	31.87	1
0	100	0	1
100	0	25.55	1

*: The percentage of additional water was calculated based on the mass of the alkaline activator used in each mixture

The dry precursor materials were first mechanically mixed to obtain a homogeneous powder blend. After that, the alkaline activator solution, together with the additional water, was gradually introduced, and mixing continued until a uniform binder was obtained. The fresh binder was

then cast into molds and lightly vibrated to eliminate entrapped air and improve compaction of the specimens.

All specimens were initially cured in a laboratory oven at 100 °C for 24 h. After this initial curing period, the specimens were demolded and divided into two groups. The first group was stored under laboratory conditions until the 28-day testing age and served as the oven-cured control. The second group was subjected to supplementary microwave treatment using a kitchen-type microwave oven set to 450 W. The microwave treatment consisted of six successive cycles, each comprising 15 s of microwave irradiation followed by 45 s of cooling at room temperature, corresponding to a total irradiation time of 90 s and an overall treatment duration of 6 min. Following microwave treatment, these specimens were stored under the same laboratory conditions as the oven-cured control specimens until the 28-day testing age.

After curing and storage, the specimens were characterized to evaluate the effects of precursor composition and supplementary microwave treatment on their mechanical and microstructural properties. The 28-day compressive strength was determined using 50 × 50 × 50 mm cube specimens in accordance with ASTM C109. Phase characteristics were identified by X-ray diffraction

(XRD), while Fourier transform infrared spectroscopy (FTIR) was employed to investigate the formation of geopolymeric reaction products and the evolution of chemical bonds. The morphology, microstructure, and elemental composition of the hardened binders were examined using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS). For the evaluation of mass loss associated with supplementary microwave treatment, the specimens were weighed after the initial 24 h oven-curing period and again after microwave treatment. The percentage mass loss was calculated from these two measurements.

3. Results and Discussion

3.1. XRD Analysis

Figure 1 presents the XRD patterns of the raw precursors and AAMs prepared with different FA/GGBFS ratios and subjected to oven curing alone or oven curing followed by supplementary microwave treatment. The raw FA exhibited several sharp diffraction peaks superimposed on a broad amorphous hump, indicating the coexistence of crystalline and amorphous phases. In contrast, raw GGBFS exhibited a predominantly amorphous structure with only weak crystalline reflections, suggesting a higher glassy content and, consequently, greater reactivity during alkali activation.

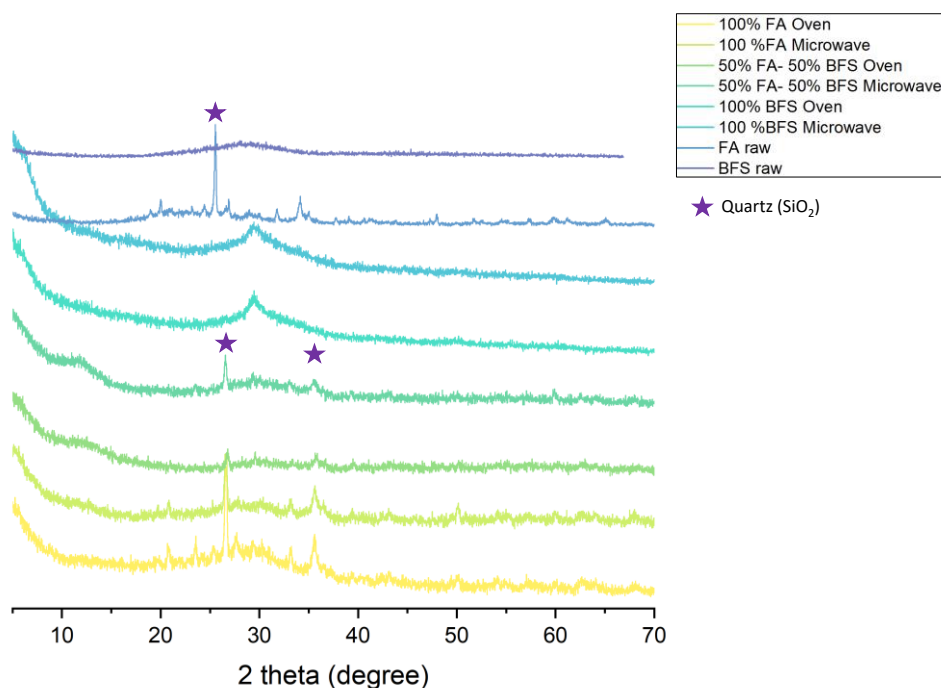


Figure 1. XRD patterns of raw FA and GGBFS precursors and alkali-activated FA/GGBFS binders with different precursor ratios subjected to oven curing alone or oven curing followed by supplementary microwave treatment.

For the 100% FA mixtures, the diffraction patterns obtained for the oven-cured control and microwave-treated specimens retained relatively pronounced crystalline reflections inherited from the fly ash

precursor. The broad amorphous hump observed between approximately 20° and 35° (2θ) became slightly more evident after activation, consistent with changes in the amorphous phase associated with alkali activation.

However, the persistence of distinct crystalline peaks suggests that a considerable fraction of the fly ash remained unreacted after curing. Only minor differences were observed between the oven-cured control and microwave-treated specimens, suggesting that supplementary microwave treatment did not substantially alter the mineralogical characteristics of the reaction products under the investigated conditions.

The 50% FA-50% GGBFS mixture exhibited intermediate characteristics between the two single-precursor systems. Compared with the 100% FA mixture, the diffraction patterns showed a broader amorphous hump and a reduced relative intensity of the crystalline reflections, consistent with the increasing contribution of the predominantly amorphous GGBFS precursor and its alkali-activated reaction products. The similarity of the diffraction patterns obtained for the oven-cured control and microwave-treated specimens suggests that supplementary microwave treatment did not substantially modify the observed phase characteristics.

For the 100% GGBFS mixtures, the XRD patterns were dominated by a broad diffuse hump, consistent with the predominantly amorphous character of the GGBFS precursor and its alkali-activated reaction products. Compared with the FA-containing mixtures, considerably weaker crystalline reflections were observed. The oven-cured control and microwave-treated specimens exhibited very similar diffraction patterns and no new crystalline phases. Overall, the XRD results indicate that

the FA/GGBFS ratio had a more pronounced influence on the observed phase characteristics than supplementary microwave treatment. Increasing the GGBFS content was associated with a more pronounced amorphous characteristics and a broader diffuse halo, whereas no substantial differences in the crystalline phase assemblage were observed between the oven-cured control and microwave-treated specimens.

3.2. FTIR Analysis

Figure 2 presents the FTIR spectra of the raw FA, raw GGBFS, and alkali-activated binders containing different FA/GGBFS ratios subjected to oven curing alone or oven curing followed by supplementary microwave treatment. Considerable differences were observed between the raw precursors and the alkali-activated specimens, indicating changes in the chemical bonding environment of the aluminosilicate materials following alkali activation.

A broad absorption band centered at approximately $3400\text{--}3500\text{ cm}^{-1}$ was observed in all specimens and is assigned to the stretching vibration of hydroxyl (O-H) groups associated with physically adsorbed and chemically bound water. The absorption band near 1640 cm^{-1} corresponds to the bending vibration of H-O-H groups, indicating the presence of water molecules retained within the reaction products. These bands are characteristic of alkali-activated binders and reflect the incorporation of water into the developing gel network during geopolymerization.

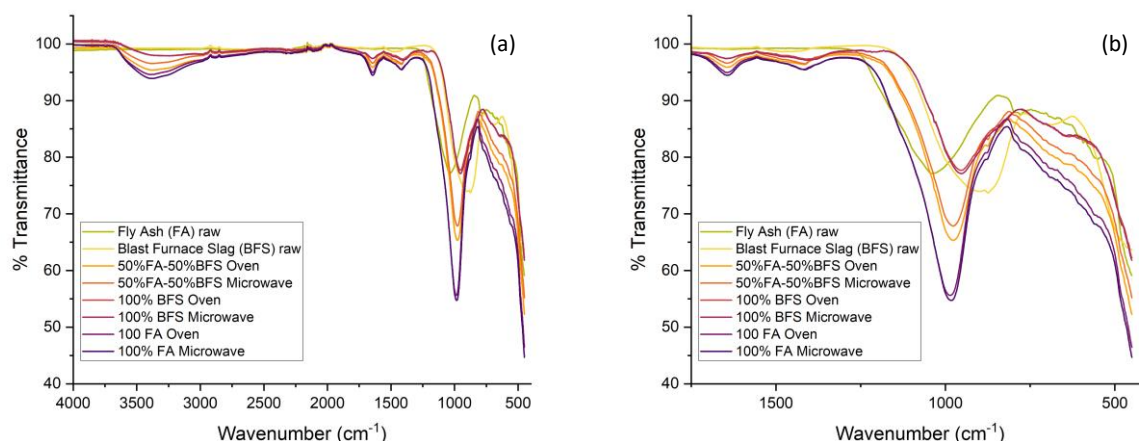


Figure 2. FTIR spectra of raw FA, raw GGBFS, and alkali-activated FA/GGBFS binders subjected to oven curing alone or oven curing followed by supplementary microwave treatment: (a) full spectra ($4000\text{--}400\text{ cm}^{-1}$) and (b) enlarged view of the fingerprint region ($1800\text{--}400\text{ cm}^{-1}$).

The most significant changes occurred within the $900\text{--}1000\text{ cm}^{-1}$ region, which corresponds to the asymmetric stretching vibration of Si-O-T (T = Si or Al) bonds and is generally regarded as the characteristic fingerprint of geopolymeric reaction products. Compared with the raw

materials, the activated specimens exhibited clear changes in both the position and shape of this absorption band, consistent with changes in the local Si-O-T bonding environment associated with alkali activation. For the 100% FA mixtures, the main Si-O-T absorption band

remained relatively sharp. Although geopolymerization was evident, the spectral features indicate that part of the original crystalline fly ash structure was retained, which is consistent with the relatively lower reactivity of FA under alkaline activation. The spectra obtained for the oven-cured control and microwave-treated specimens exhibited very similar absorption bands, with no new functional groups, indicating that supplementary microwave treatment did not produce detectable changes in the principal chemical functionalities within the resolution of the FTIR analysis.

In contrast, the 50% FA-50% GGBFS mixtures exhibited a broader and slightly modified Si-O-T absorption band compared with the FA-rich system. Compared with the FA-rich mixtures, the broadening of the Si-O-T band indicates a change in the local bonding environment associated with the incorporation of GGBFS. Such behavior has frequently been observed in blended alkali-activated systems, where calcium contributes to the formation of a denser reaction network.

For the 100% GGBFS mixtures, the Si-O-T band became noticeably broader than that of the FA-rich specimens, indicating a different chemical bonding environment compared with the FA-rich mixtures. The absorption bands below approximately 600 cm^{-1} , assigned to the bending vibrations of Si-O-Si and Si-O-Al linkages, were also clearly observed, further confirming the development of a continuous aluminosilicate framework following alkali activation.

Comparison of the two treatment conditions revealed that the overall FTIR spectra of the oven-cured control and microwave-treated specimens were highly similar across all precursor compositions. No additional absorption bands or substantial shifts in the characteristic Si-O-T region were observed after supplementary microwave treatment, indicating that microwave irradiation did not introduce any detectable new chemical functionalities within the resolution of the FTIR analysis.

3.3. SEM Analysis

SEM micrographs (Figures 3 and 4) revealed distinct morphological differences depending on both the precursor composition and the supplementary microwave treatment. The GGBFS-containing specimens exhibited visible crack formation in both the oven-cured control and microwave-treated groups. However, the cracks observed in the microwave-treated GGBFS-based specimens were wider and more pronounced than those in the oven-cured counterparts (Figure 3a, 3b, 4a, and 4b). This may be associated with rapid internal heating and

moisture evaporation during microwave treatment, which can contribute to internal stresses within the hardened matrix.

In contrast, the 100% FA specimens showed fewer visible cracks than the GGBFS-containing mixtures. However, numerous spherical fly ash particles were clearly observed, indicating the presence of partially reacted or unreacted FA particles within the matrix (Figure 3c, 3d, 4c, and 4d). The persistence of these spherical particles suggests that FA underwent a lower degree of reaction under the applied curing conditions than GGBFS.

The 50% FA-50% GGBFS mixtures displayed a more complex and heterogeneous morphology, reflecting the combined contribution of both precursors. With the incorporation of GGBFS, crack formation was again observed, particularly in the microwave-treated specimen, where cracks appeared more evident. In addition, a relatively porous microstructure was detected in the blended mixtures. This porous morphology may be related to the presence of unreacted FA particles and the heterogeneous packing/reaction behavior of the FA-GGBFS system (Figure 3e, 3f, 4e, and 4f).

As a result, the SEM observations indicate that GGBFS incorporation was associated with increased crack formation, especially following supplementary microwave treatment, whereas FA-rich mixtures retained more unreacted spherical particles and exhibited a more porous morphology. These findings suggest that precursor composition and supplementary microwave treatment both contributed to differences in the microstructural development of the alkali-activated binders.

To further investigate the effect of precursor composition on the chemical characteristics of the reaction products, the atomic ratios obtained from EDS analysis were evaluated using the Ca-Al-Si ternary diagram and the Ca/Si versus Si/Al diagrams (Figure 5a and Figure 5b). The Ca-Al-Si ternary diagram reveals a clear separation among the three mixtures based on their precursor compositions. The 100% GGBFS specimens are positioned closer to the Ca-rich region, reflecting the higher calcium content inherited from GGBFS. In contrast, the 100% FA specimens are located toward the Si-Al-rich region, consistent with the aluminosilicate-rich composition of FA. The 50% FA-50% GGBFS mixtures occupy an intermediate position between the two end-member systems, indicating that incorporating GGBFS modifies the elemental balance of the analyzed regions while maintaining contributions from both precursors.

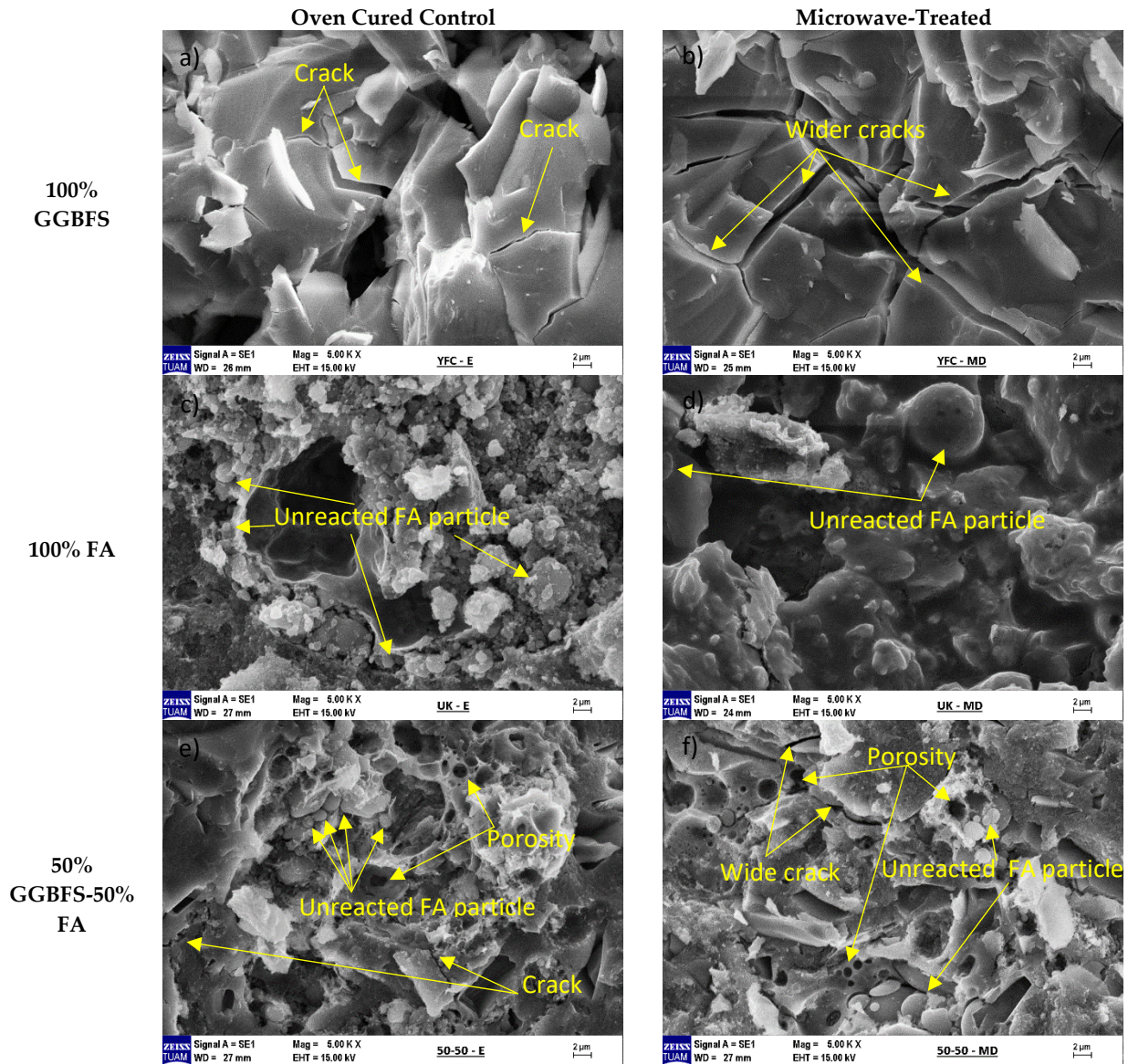


Figure 3. SEM micrographs of alkali-activated binders at 5000× magnification containing (a-b) 100% GGBFS, (c-d) 100% FA, and (e-f) 50% GGBFS-50% FA for the oven-cured control and microwave-treated specimens, respectively, highlighting surface morphology and microstructural features.

The Ca/Si versus Si/Al plot further demonstrates the compositional differences among the investigated mixtures. The 100% GGBFS specimens exhibited the highest Ca/Si ratios (0.55-0.62) together with the highest Si/Al ratios (3.6-3.8). Conversely, the 100% FA specimens showed substantially lower Ca/Si ratios (0.07-0.12) and lower Si/Al ratios (3.0-3.1). The 50% FA-50% GGBFS mixtures displayed intermediate values, confirming that the elemental composition gradually changed with increasing GGBFS incorporation. These results indicate that increasing the GGBFS content systematically increased the relative calcium concentration within the analyzed regions, whereas FA-rich specimens remained dominated by silicon and aluminum. The intermediate position of the blended mixtures suggests that both precursors contributed to the elemental composition of the analyzed regions

3.4. Compressive Strength

The compressive strength results for AAMs prepared with different FA/GGBFS ratios and subjected to oven curing alone or to oven curing followed by supplementary microwave treatment are presented in Figure 6a. The results demonstrate that both the precursor composition and supplementary microwave treatment influenced the 28-day mechanical performance of the binders. Among all mixtures, the 50% FA-50% GGBFS specimens exhibited the highest compressive strength under both treatment conditions, reaching approximately 14.2 MPa for the oven-cured control and 11.2 MPa for the microwave-treated specimens. The higher mechanical performance of the blended system suggests that the combined use of FA and GGBFS provided a more favorable chemical composition for the development of alkali-activated reaction products than either precursor used individually.

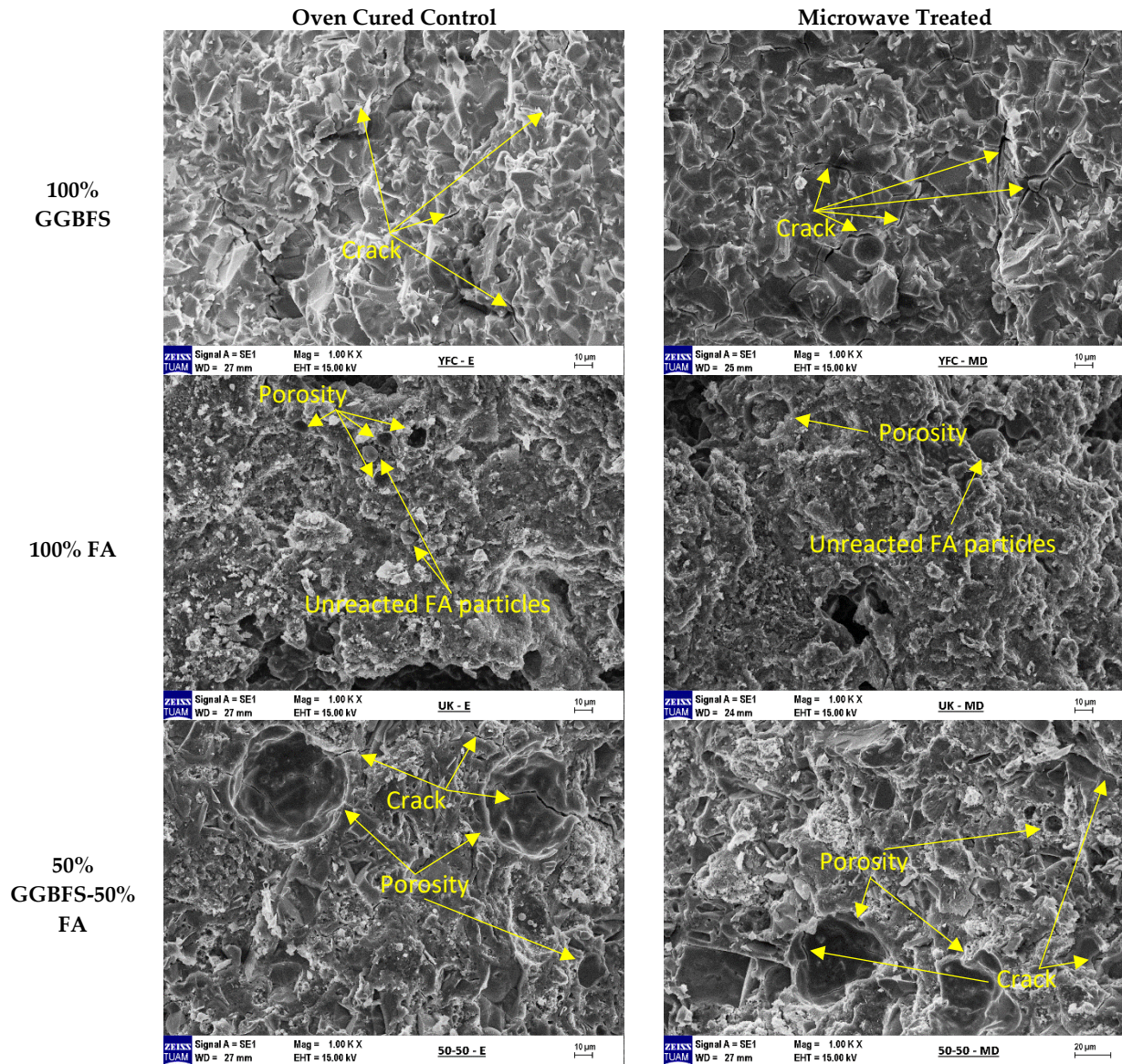


Figure 4. SEM micrographs of alkali-activated binders at 1000× magnification containing (a-b) 100% GGBFS, (c-d) 100% FA, and (e-f) 50% GGBFS-50% FA for the oven-cured control and microwave-treated specimens, respectively.

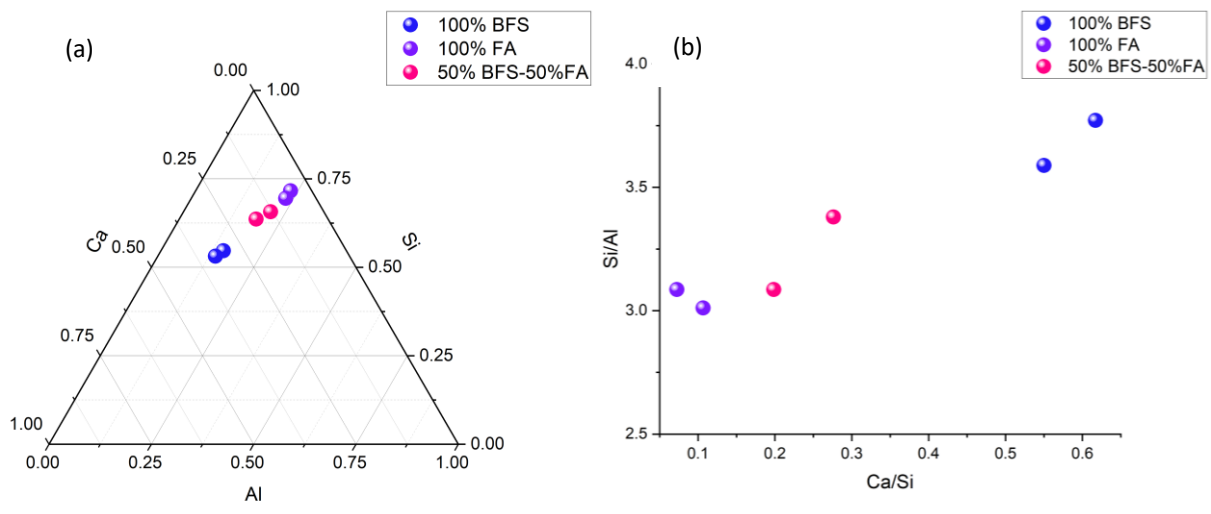


Figure 5. SEM-EDS results of alkali-activated binders containing different FA/GGBFS ratios: (a) Ca-Al-Si ternary diagram showing the elemental distribution and (b) relationship between Si/Al and Ca/Si atomic ratios.

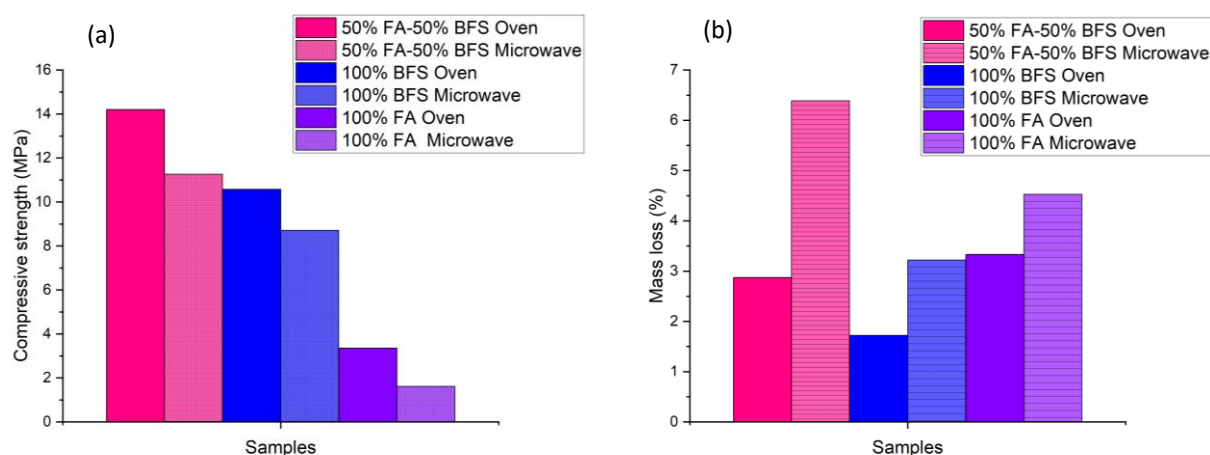


Figure 6. Effect of precursor composition and supplementary microwave treatment on the properties of alkali-activated binders. a) 28-day compressive strength and b) mass loss of specimens prepared with different FA/GGBFS ratios for the oven-cured control and microwave-treated groups.

In contrast, the 100% GGBFS specimens achieved intermediate compressive strengths of approximately 10.5 MPa and 8.7 MPa for the oven-cured control and microwave-treated specimens, respectively, whereas the 100% FA mixtures exhibited the lowest strength values, remaining below 4 MPa under both treatment conditions. Although the measured compressive strengths are lower than those reported for some heat-cured AAM systems, direct comparison is difficult because mechanical performance strongly depends on precursor characteristics, activator composition, curing temperature, liquid-to-solid ratio, and specimen preparation.

The significantly lower strength of the FA-rich specimens is consistent with the SEM observations, where numerous unreacted spherical fly ash particles remained embedded within the hardened matrix (Figure 3c, 3d, 4c, and 4d). The persistence of these particles is consistent with incomplete reaction of the FA precursor and, consequently, the development of a less continuous reaction matrix. In contrast, the incorporation of GGBFS resulted in a more continuous reaction matrix, although microcracks became evident in both oven-cured control and microwave-treated specimens. These cracks were particularly pronounced in the microwave-treated GGBFS-containing mixtures and may have reduced the load-bearing capacity of the hardened matrix despite the higher intrinsic reactivity of GGBFS.

FTIR analysis further supports the occurrence of alkali activation in all mixtures through the presence of the characteristic Si-O-T (T = Si or Al) absorption band (Figure 2). While comparable functional groups were detected regardless of the supplementary microwave treatment, differences in precursor composition modified the chemical bonding environment, particularly within the

principal aluminosilicate vibration region. Moreover, SEM-EDS analysis revealed a systematic increase in the Ca/Si ratio with increasing GGBFS content, whereas the FA-rich mixtures were characterized by considerably lower calcium contents. This compositional evolution is consistent with the different reaction mechanisms expected for the two precursors. FA-rich systems are generally dominated by sodium aluminosilicate hydrate (N-A-S-H) type gels, whereas the incorporation of GGBFS introduces calcium into the reaction system, favoring the formation of calcium-containing aluminosilicate hydrates (C-(A)-S-H) together with hybrid aluminosilicate binding phases. Although the present analytical techniques do not allow direct identification or quantification of these gels, the observed elemental compositions are consistent with this well-established reaction mechanism reported in the literature (Liang et al., 2024; Pawluczuk et al., 2021; Wang et al., 2024).

Supplementary microwave treatment following initial oven curing was associated with lower 28-day compressive strength for all precursor compositions compared with the corresponding oven-cured controls. Since the FTIR spectra remained generally comparable between the two treatment conditions, the reduction in strength cannot be directly attributed to major differences in the detectable chemical functionalities. SEM observations showed wider and more pronounced cracks in the microwave-treated GGBFS-containing specimens (see Figure 3 and 4). In addition, the greater mass loss observed in the microwave-treated specimens suggests more intensive moisture removal due to the additional microwave exposure. The combined SEM and mass-loss results suggest that rapid moisture removal during supplementary microwave treatment may have contributed to the development of internal stress and microcrack formation, thereby adversely affecting the 28-

day mechanical performance. However, because internal temperature, vapor pressure, and stress development were not measured directly, this mechanism should be regarded as a plausible interpretation rather than a demonstrated process.

To sum up, the findings demonstrate that the precursor composition exerted a greater influence on compressive strength than the supplementary microwave treatment under the conditions investigated. The blended 50% FA–50% GGBFS system exhibited the best mechanical performance, indicating a beneficial interaction between the two precursors, whereas supplementary microwave treatment following initial oven curing, under the applied curing conditions, resulted in lower compressive strengths than the oven-cured control.

4. Conclusions

The influence of precursor composition and supplementary microwave treatment, following initial oven curing, on the microstructural characteristics and 28-day mechanical performance of alkali-activated FA/GGBFS binders was systematically investigated. The results demonstrated that the precursor composition played a more pronounced role in the properties of the alkali-activated binders than the supplementary microwave treatment. Increasing the GGBFS content was associated with more predominantly amorphous phase characteristics, whereas FA-rich mixtures retained more crystalline features inherited from the raw fly ash. FTIR analysis showed that alkali activation modified the chemical bonding environment through changes in the characteristic Si–O–T absorption band, while comparable functional groups were observed for the oven-cured control and microwave-treated specimens, indicating that supplementary microwave treatment did not alter the fundamental types of chemical bonds formed. SEM observations revealed clear differences in microstructure, with GGBFS-containing specimens exhibiting a more continuous reaction matrix but also more pronounced crack formation, particularly following supplementary microwave treatment. In contrast, FA-rich specimens contained numerous unreacted spherical FA particles, reflecting their lower reactivity under the applied curing conditions, whereas the blended FA–GGBFS mixtures displayed a more heterogeneous morphology. SEM–EDS analysis further showed a gradual increase in the Ca/Si ratio with increasing GGBFS content, while FA-rich mixtures remained dominated by Si and Al. This compositional evolution is consistent with a gradual transition from Si- and Al-rich reaction products in FA-rich systems to increasingly Ca-containing alkali-activated

binding phases as GGBFS content increases. Among all mixtures, the 50% FA–50% GGBFS binder exhibited the highest compressive strength, reaching 14.2 MPa for the oven-cured control and 11.2 MPa for the microwave-treated specimens, suggesting a beneficial combined effect of FA and GGBFS under the investigated conditions.

Supplementary microwave treatment following initial oven curing resulted in lower 28-day compressive strength than oven curing alone for all investigated precursor compositions. The greater mass loss and more pronounced microcrack formation observed in the microwave-treated specimens suggest that rapid moisture removal during microwave exposure may have contributed to microstructural damage and the subsequent reduction in mechanical performance. Under the investigated conditions (450 W, six heating-cooling cycles), supplementary microwave treatment therefore provided no mechanical advantage over the oven-cured control at 28 days. However, the present experimental design does not allow conclusions to be drawn regarding whether microwave treatment can accelerate early-age strength development or reduce the time required to achieve a target compressive strength, since both groups were mechanically tested at 28 days. Future studies should therefore evaluate compressive strength immediately after microwave treatment and at selected early curing ages and compare these results with the subsequent strength development of oven-cured controls. Such investigations would clarify whether optimized microwave treatment can effectively accelerate strength development while limiting moisture-induced microcracking.

Declaration of Ethical Standards

The authors declare that they complied with all ethical standards throughout the preparation and publication of this study.

ORCID iD Authorship Contribution Statement

Author-1: Investigation, Formal analysis, Data curation, Writing-original draft.

Author-2: Investigation, Formal analysis, Data curation, Writing-original draft.

Author-3: Conceptualization, Methodology, Formal analysis, Writing-review and editing, Funding.

Author-4: Conceptualization, Methodology, Formal analysis, Supervision, Visualization, Writing-original draft, Writing-review and editing, Funding.

Declaration of Competing Interest

The authors have no conflicts of interest to declare regarding the content of this article.

Data Availability Statement

The datasets supporting the findings of this study are available from the corresponding author upon reasonable request.

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