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Reaction kinetics and microstructural evolution of alkali-activated electric arc furnace slag

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Abstract:

Electric arc furnace slag (EAFS) is a steelmaking by-product with potential for use as a precursor in low-clinker or clinker-free cementitious materials. However, the reaction mechanisms governing its alkali activation, particularly in Fe-rich systems, remain insufficiently understood. This study investigates the alkali activation of EAFS as the sole precursor using sodium- and potassium-based activating solutions with different silica moduli and alkali contents. The results show that activator chemistry strongly influenced early reaction kinetics, setting behaviour and mechanical performance. Sodium-activated systems generally exhibited higher reactivity and greater compressive strength, whereas potassium-activated mixtures showed slower but sustained strength development. Semi-adiabatic calorimetry indicated that early-age behaviour was mainly governed by rapid precipitation processes, followed by progressive structural reorganisation during curing. FTIR analysis revealed a gradual shift of the main Si–O–T band towards lower wavenumbers, suggesting changes in the bonding environment and continued gel evolution. Electrical resistivity—used as an indirect indicator of pore network connectivity—and drying shrinkage were not controlled solely by total open porosity or mass loss, but were strongly affected by gel organisation. Overall, the study provides an integrated interpretation of how reaction kinetics, gel evolution and the developing matrix structure govern the performance of alkali-activated EAFS systems.

1 Introduction

Electric arc furnace slag (EAFS) is one of the main solid by-products generated during steel production and is produced in large quantities worldwide. Due to its high content of calcium, iron, and aluminosilicate phases, EAFS has attracted increasing attention as a potential precursor for alkali-activated materials (AAMs), offering an alternative pathway for the valorisation of industrial waste and the reduction of the environmental impact associated with conventional Portland cement production. In addition to its environmental benefits, the use of EAFS in alkali-activated systems may contribute to the development of circular economy strategies through the reuse of low-value industrial residues in high-performance construction materials [1], [2].

From a scientific and technical standpoint, alkali-activated materials are produced through the reaction between a solid aluminosilicate precursor and an alkaline activating solution. The most commonly used activators include hydroxides, silicates, carbonates, and sulphates. Kassim et al. [3] investigated the influence of CO_2 on mortars produced with alkali-activated EAFS using different $\text{NaOH}/\text{Na}_2\text{SiO}_3$ ratios, while Gómez-Casero et al. [4] analysed the microstructure of binders based on ferrous and non-ferrous slags activated with KOH and K_2SiO_3 . Other authors, such as Tan et al. [5], employed Na_2CO_3 and Na_2SO_4 as activators for blast furnace slag systems.

The type and dosage of the alkaline activator play a critical role in slag activation, as they directly control the alkalinity of the pore solution and, consequently, the dissolution kinetics of the precursor. Sodium hydroxide (NaOH) generates highly alkaline environments that promote rapid dissolution of reactive phases and accelerate the early formation of reaction products. In contrast, sodium silicate (Na_2SiO_3) acts not only as an activator but also as a source of soluble silicate species, directly contributing to the formation of calcium–aluminosilicate hydrate type gels. Conversely, activators such as sodium carbonate (Na_2CO_3) and sodium sulphate (Na_2SO_4), due to their lower effective alkalinity, tend to delay precursor dissolution and retard the onset of reaction processes. Therefore, activation efficiency depends not only on alkali concentration but also on the chemical nature of the activator and parameters such as silica modulus (M_s) and alkali content, which govern both nucleation and the growth of reaction products [6], [7], [8].

The alkali activation of calcium-rich aluminosilicate materials such as EAFS involves a sequence of physicochemical processes that determine the nature and properties of the resulting binding structure. Under highly alkaline conditions, dissolution of the precursor releases silicon, aluminium, and calcium species into solution. These species subsequently undergo condensation and precipitation reactions, leading to the formation of calcium–aluminosilicate hydrate type gels, commonly described as C-A-S-H or C-(N)-A-S-H in high-calcium systems [6], [7], [9], [10], [11]. The composition and organisation of these gels strongly influence the mechanical performance, transport properties, and long-term stability of alkali-activated materials.

A particularly relevant aspect of EAFS is its high iron content and the potential role of this element during alkali activation. Previous studies have suggested that part of the Fe initially present in the precursor may participate in the reaction products after partial oxidation from Fe^{2+} to Fe^{3+} . Lemougna et al. [12], using Mössbauer spectroscopy, showed that Fe species can undergo changes in oxidation state and coordination environment during alkali activation, while Eliche et al. [4] reported the possible formation of C-(Fe)-A-S-H type gels in Fe- and Ca-rich slag systems. Mahajan et al. [13] observed the incorporation of Fe into alkali-activated copper slag systems. Although the exact mechanism of Fe incorporation into alkali-activated binding

structures remains uncertain, previous studies suggest that Fe may influence the structural organisation and long-term evolution of the gel network [14].

The chemical and mineralogical composition of EAFS differs considerably from that of more widely studied precursors such as blast furnace slag or fly ash. In particular, EAFS is characterised by a comparatively lower amorphous content and a high concentration of Fe-bearing phases, factors that may strongly influence its reactivity and the evolution of the reaction products formed during alkali activation. Despite these characteristics, most published research has mainly focused on the partial incorporation of EAFS as a supplementary precursor combined with materials such as blast furnace slag, fly ash, or Portland cement [10], [11], [15], [16]. In contrast, studies investigating EAFS as the main or sole precursor remain relatively limited.

Furthermore, most available studies are primarily focused on compressive strength development and phase identification at early curing ages, commonly limited to 28 days [17], [18], [19], [20]. Although these investigations provide valuable information regarding the feasibility of alkali-activated EAFS systems, the long-term microstructural evolution and transport behaviour remain insufficiently understood. This aspect is particularly relevant considering that gel reorganisation, pore network refinement, and transport properties may continue evolving well beyond the early curing stages [21].

Another important aspect is related to the industrial valorisation of the material itself. The fine EAFS fraction used in this study (0/5 mm) is generated as an unavoidable by-product during the production of other slag fractions with higher commercial demand. As a result, this fine fraction often presents limited applications and low commercial value despite being generated in significant quantities. In this context, the development of high-value applications based on the complete alkali activation of this material may contribute to improving waste valorisation, resource efficiency, and the overall sustainability of steel slag management strategies.

In addition, the relationship between reaction kinetics, microstructural evolution, ionic and fluid transport behaviour, and dimensional stability in Fe-rich alkali-activated systems remains poorly understood. While previous studies have generally focused on isolated properties, limited attention has been given to the integrated interpretation of calorimetric response, gel evolution, pore network development, electrical resistivity, and drying shrinkage behaviour. Understanding these interactions is essential for explaining the long-term performance of alkali-activated systems and for optimising their formulation.

In this context, the present study investigates the alkali activation of EAFS used as the sole precursor by means of different sodium- and potassium-based activating solutions with varying silica modulus and alkali contents. Particular attention is given to the relationship between reaction kinetics, microstructural evolution, ionic transport properties, and dimensional stability up to 120 days of curing. To this end, semi-adiabatic calorimetry, setting time analysis, compressive strength, electrical resistivity, open porosity, drying shrinkage, mass loss, and FTIR analyses were combined in order to establish an integrated interpretation of the mechanisms governing the behaviour of the investigated systems.

2 Materials and Methods

2.1 Characterisation of materials

In this study, a slag derived from steel production by means of an electric arc furnace (EAFS) was used as the precursor material. The material, with an original particle size fraction of 0–5 mm, was subjected to a grinding process in order to reduce its particle size and increase its

reactivity. Laser diffraction particle size analysis (Mastersizer 2000, Malvern Instruments, Malvern, United Kingdom) yielded a d50 of 39.8 μm , with d10 and d90 values of 4.7 μm and 279.1 μm , respectively.

X-ray fluorescence (XRF) analysis performed using a Philips PW2400 spectrometer (Philips Analytical, Almelo, The Netherlands), revealed that the EAFS can be classified as a calcium-rich ferro-aluminosilicate material, with Fe_2O_3 (35.1%), CaO (22.3%), Al_2O_3 (15.0%), and SiO_2 (14.8%) as its main constituents. The chemical composition of the EAFS is summarised in Table 1

Table 1. Chemical properties of EAFS (%)

Material	Fe_2O_3	CaO	Al_2O_3	SiO_2	MnO	Cr_2O_3	MgO	SO_3	Others	LOI
EAFS	35.11	22.26	15.00	14.84	5.64	2.39	2.28	0.33	1.85	0.06

The density of the precursor material was determined following the procedures established in UNE 80103:2013 [22]. The 0/4 mm fraction of limestone sand was used to manufacture mortars. For this aggregate, the particle density and water absorption were determined in accordance with UNE-EN 1097-6 [23]. The tests yielded a density of 3.71 g/cm^3 for the EAFS, while the sand exhibited an apparent particle density of 2.68 g/cm^3 , an oven-dry particle density of 2.63 g/cm^3 , and a water absorption of 0.77%.

The mineralogical composition of the precursor was determined by X-ray diffraction (see Section 2.2 for equipment description and testing parameters). The main crystalline phases identified in the diffraction pattern were gehlenite, wuestite, calcite, and magnetite. Using the Rietveld method with alumina ($\alpha\text{-Al}_2\text{O}_3$) as an internal standard, the precursor material was estimated to consist of 58.6% crystalline phases and 41.4% amorphous phases. Figure 1(a) shows the main crystalline phases detected in the XRD spectrum.

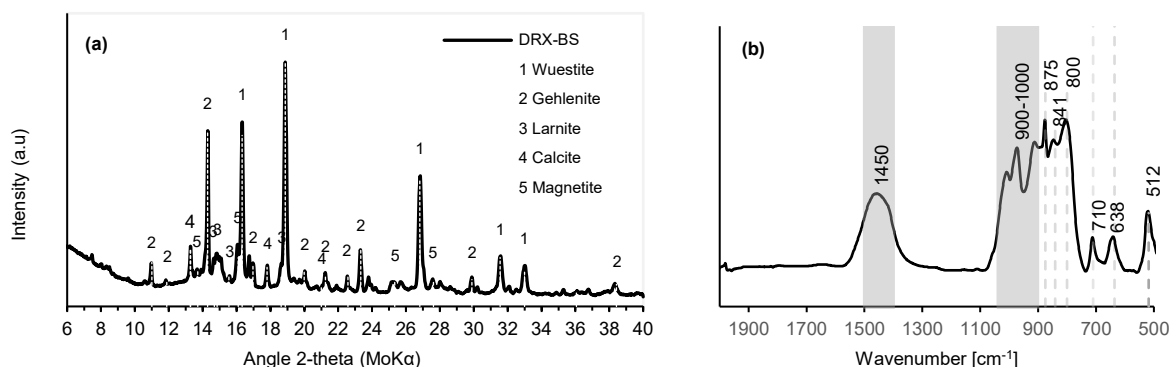


Figure 1. Diffractograms of EAFS (a) and FTIR of EAFS (b).

To identify the functional groups of the precursor, Fourier-transform infrared (FTIR) spectroscopy was performed (see Section 2.2 for equipment description and testing parameters).

The FTIR spectrum of the precursor (Figure 1b) exhibits the characteristic behaviour of this type of material, with intense bands associated with silicate and aluminosilicate networks. The signals at 638 cm^{-1} and 512 cm^{-1} are attributed to metal–oxygen bond vibrations; in particular, the peak centred at 512 cm^{-1} can be associated with the Fe–O vibration corresponding to the presence of wuestite (FeO), in agreement with XRD results [24], [25].

The peak centred at 710 cm^{-1} corresponds to the bending vibration of the C–O bond associated with the carbonate ion (CO_3^{2-}) present in calcite [24], [26], while the band at 800 cm^{-1} is related

to the bending vibration of the Al–O–Si bond, characteristic of aluminosilicate structures [26]. The signal observed at 841 cm^{-1} is associated with vibrations of the SiO_4^{4-} tetrahedron corresponding to β -dicalcium silicate (Ca_2SiO_4), also known as larnite.

The band around 875 cm^{-1} is mainly attributed to vibrations of the carbonate ion (CO_3^{2-}); however, given the high iron content in the slag (35.11 % Fe_2O_3) and the mineralogical presence of Fe-rich phases, such as wuestite (19 %) and magnetite (3.6 %), this signal may also include secondary contributions from Fe–O bond vibrations, associated with distortions of iron oxides within the silicate matrix.

The broad and intense band observed between 900 cm^{-1} and 1000 cm^{-1} is attributed to asymmetric stretching vibrations of the Si–O–T bond (where T = Si or Al), which are characteristic of silicate and aluminosilicate networks, indicating that these phases constitute predominant components of the EAFS [4], [24]. Finally, the band centred at 1450 cm^{-1} corresponds to the vibrational modes of the carbonate ion (CO_3^{2-}).

Alkali activators consisted of sodium- and potassium-based solutions prepared by combining sodium hydroxide (NaOH) or potassium hydroxide (KOH) with the corresponding silicate solution, namely sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3). The sodium silicate solution, also referred to as water glass, had the following composition: $\text{SiO}_2 = 26.4\%$, $\text{Na}_2\text{O} = 8.0\%$ and $\text{H}_2\text{O} = 65.6\%$, whereas the potassium silicate solution consisted of $\text{SiO}_2 = 23.0\%$, $\text{K}_2\text{O} = 11.0\%$ and $\text{H}_2\text{O} = 66.0\%$. All reagents were commercially available.

2.2 Methods

2.2.1 Testing on Pastes

Compressive strength tests were carried out on $25 \times 25 \times 25\text{ mm}$ cubic specimens, using a Zwick/Roell testing machine (ZwickRoell GmbH & Co., Ulm, Germany), adapting the procedures outlined in the UNE-EN 196-1 standard [27].

The initial and final setting times were determined following the methodology described in UNE-EN 196-3:2017 [28].

Temperature evolution in the selected pastes was monitored using semi-adiabatic equipment. The solid and liquid components were mixed for 4 minutes in order to minimise the loss of information associated with the early-stage reactions. The temperature increase resulting from the exothermic reactions was recorded using thermocouples embedded in the pastes and connected to a data acquisition system.

For microstructural analysis of the pastes, the reaction was stopped using the solvent exchange method [29]. The samples were then ground to a particle size of $<0.063\text{ mm}$ prior to FTIR and XRD analysis.

X-ray diffraction (XRD) analysis was performed on the raw EAFS precursor using a Bruker D8 Advance diffractometer (Bruker AXS GmbH, Karlsruhe, Germany), equipped with a $\text{MoK}\alpha$ radiation source (50 kV and 50 mA). Scans were conducted in the range of 3.5° to 35° (2θ), with continuous sample rotation at 10 rpm. Phase identification was carried out using DIFFRAC EVA V5.2 software. XRD analysis was used to identify the main crystalline phases of the precursor and to estimate its crystalline and amorphous fractions by the Rietveld method.

Fourier transform infrared spectroscopy (FTIR) was used to identify the functional groups of the EAFS precursor and the selected alkali-activated pastes. A PerkinElmer Spectrum Two

spectrometer was employed over the range 4000–400 cm^{-1} . Each measurement consisted of 32 scans with a resolution of 4 cm^{-1} .

2.2.2 Testing on Mortars

The compressive strength of the mortars was determined using $40 \times 40 \times 160$ mm prismatic specimens, previously tested in flexure, in accordance with UNE-EN 196-1 [27]. The tests were carried out using a Zwick/Roell testing machine (ZwickRoell GmbH & Co., Ulm, Germany).

The bulk density, water absorption and open porosity of the mortars were determined in accordance with ASTM C 642-13 [30]. In addition, electrical resistivity was measured as an indirect indicator of pore network connectivity using a Resipod surface resistivity meter (Proceq SA, Schwerzenbach, Switzerland).

Prismatic specimens measuring $25 \times 25 \times 250$ mm were prepared in accordance with UNE-EN 196-1:2018 [27] for drying shrinkage measurements, which were conducted following ASTM C490-04 [31]. For each mortar formulation, four specimens were produced: two were stored in a humidity chamber (95% relative humidity, 21 °C) and two under controlled ambient conditions (60% relative humidity, 21 °C). Measurements were performed using a Sylvac S229 electronic micrometer (Sylvac SA, Le Landeron, Switzerland). In addition, mass variation due to drying was monitored by periodically weighing the same specimens used for the shrinkage test using a digital balance.

2.3 Experimental procedures

The research methodology was carried out in two main stages.

Stage 1

Once the materials had been characterised, the alkaline activating solutions were prepared 24 hours prior to use. The different dosages employed are presented in Table 2 and Table 3 for sodium- and potassium-based activators, respectively.

The liquid-to-solid ratio (L/S) was fixed at 0.25 for all mixtures, where “L” represents the sum of the added mixing water and the water provided by the silicate solution, and “S” corresponds to the total solid content, including the precursor, the alkali hydroxide and the solid fraction of the silicate solution.

Using these dosages, 12 initial pastes were prepared with sodium-based activators and 12 with potassium-based activators. Cubic specimens ($25 \times 25 \times 25$ mm) were cast and tested in compression at 7 and 28 days (Table 2 and Table 3). All specimens were cured in a humidity chamber (95% relative humidity and 21 °C) from casting until the testing age.

Stage 2

Based on the compressive strength and workability results obtained in Stage 1, the best-performing dosages were selected. New pastes and mortars were then prepared using a liquid-to-solid ratio of 0.35.

The selected pastes were subjected to setting time tests, semi-adiabatic calorimetry and FTIR analysis.

For mortars, mix proportions were defined by mass, maintaining a sand-to-precursor (EAFS) ratio of 2.8:1. The specimens were cured in a humidity chamber from casting until the testing age. The mortars were evaluated in terms of compressive strength, density, water absorption, porosity, drying shrinkage and mass variation due to drying.

3 Results and Discussion

3.1 Initial dosages

Table 2 presents the compressive strength values measured at 7 and 28 days for 25×25 mm cubic specimens corresponding to the initial pastes produced from EAFS activated with different proportions of Na_2SiO_3 and NaOH. Table 3, in turn, shows the results obtained for the pastes prepared using K_2SiO_3 and KOH.

Table 2. Compressive strength of EAFS pastes activated with Na_2SiO_3 –NaOH at 7 and 28 days (L/S = 0.25)

Type of Paste	Ms		Compressive Strength (MPa)			
	$\text{SiO}_2/\text{Na}_2\text{O}$	$\text{Na}_2\text{O}/\text{P}$	7 days	$\pm$ SD	28 days	$\pm$ SD
S1.0-3	1.0	3%	nh	nh	nh	nh
S1.0-5	1.0	5%	4.09	0.26	28.14	0.74
S1.5-3	1.5	3%	0.40	0.03	0.89	0.01
S1.5-5	1.5	5%	4.73	0.73	26.64	1.29
S1.5-6	1.5	6%	8.92	0.48	23.93	0.70
S1.85-5	1.9	5%	8.35	0.22	21.35	0.24
S2.0-3	2.0	3%	1.78	0.23	3.78	0.20
S2.0-5	2.0	5%	11.99	0.23	22.35	1.87
S2.0-6	2.0	6%	17.23	0.87	33.62	0.86
S2.5-3	2.5	3%	7.69	0.66	11.74	0.76
S2.5-5	2.5	5%	22.83	0.69	32.13	1.95
S2.5-6	2.5	6%	20.13	0.58	26.94	0.79

Table 3. Compressive strength of EAFS pastes activated with K_2SiO_3 –KOH at 7 and 28 days (L/S = 0.25)

Type of Paste	Ms		Compressive Strength (MPa)			
	$\text{SiO}_2/\text{K}_2\text{O}$	$\text{K}_2\text{O}/\text{P}$	7 days	$\pm$ SD	28 days	$\pm$ SD
P1.0-3	1.0	3%	nh	nh	0.74	0.12
P1.0-5	1.0	5%	0.60	0.01	2.00	0.02
P1.5-3	1.5	3%	0.50	0.01	1.08	0.11
P1.5-5	1.5	5%	2.19	0.02	5.11	0.64
P1.85-3	1.85	3%	1.15	0.14	1.68	0.15
P1.85-5	1.85	5%	4.98	0.09	6.10	0.11
P1.85-6	1.85	6%	8.06	0.97	8.89	1.36
P2.0-3	2.0	3%	1.44	0.10	1.85	0.14
P2.0-5	2.0	5%	12.83	0.15	21.78	1.83
P2.5-3	2.5	3%	rh	rh	rh	rh

P2.5-5	2.5	5%	rh	rh	rh	rh
P2.5-6	2.5	6%	rh	rh	rh	rh

The compressive strength reported as "nh" is attributed to the absence of hardening during the first 48 hours; consequently, those specimens were excluded from testing. In contrast, the compressive strengths reported as "rh" correspond to pastes that exhibit extremely rapid setting, which prevented their proper casting in the moulds.

Based on the compressive strength results and on the visual assessment of paste workability during mixing and casting, the formulations exhibiting the best balance between mechanical performance and a minimum workability time of 20 min were selected for further study. According to this criterion, four sodium-activated mixtures, S1.5-5, S1.85-5, S2.0-6 and S2.5-6, and two potassium-activated mixtures, P1.85-6 and P2.0-5, were selected.

Overall, sodium-activated pastes showed superior mechanical performance compared to potassium-activated systems, although this trend depended on the specific silica modulus and alkali content. The selected formulations were subsequently used to prepare both pastes and mortars, which were then subjected to the experimental procedures described in the previous section.

3.2 Reaction kinetics and setting behaviour

The semi-adiabatic calorimetry results revealed that all mixtures were characterised by a rapid increase in temperature immediately after activation, indicating that the early-age behaviour of the systems was dominated by fast precipitation reactions occurring shortly after mixing. In all cases, the main thermal peak appeared within the first hours of reaction, followed by a progressive decrease in heat evolution over time. This behaviour is consistent with the rapid formation of initial reaction products and agrees with the mechanism previously discussed for alkali-activated slag systems.

Figure 2 presents the temperature evolution of the sodium-activated and potassium-activated systems together with the corresponding setting time markers. The sodium-activated mixtures showed a clear increase in both maximum temperature and thermal response with increasing activator concentration. In particular, the S2.5-6 mixture exhibited the highest maximum temperature and the fastest kinetic evolution at early ages, whereas S1.5-5 showed the slowest response. These differences were also reflected in the setting behaviour, where higher activator concentrations led to shorter initial and final setting times [32].

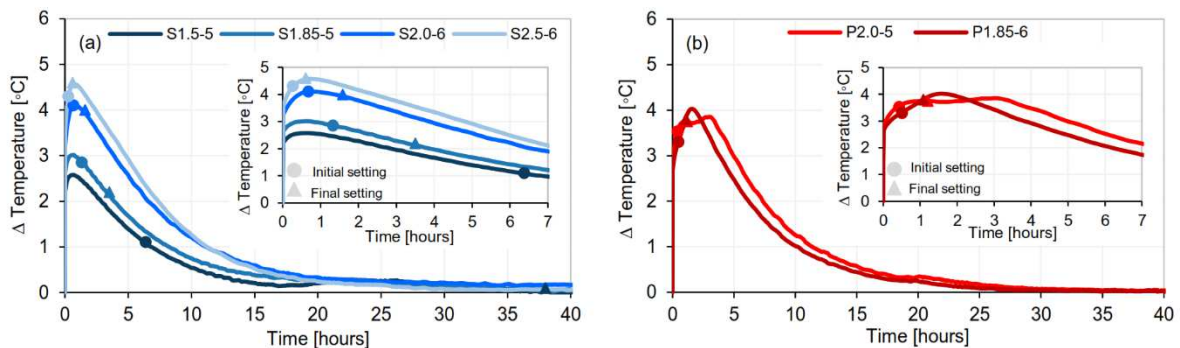


Figure 2. Semi-adiabatic temperature evolution of the selected mixtures: (a) sodium-activated systems and (b) potassium-activated systems. Insets show the early-age response and setting time markers.

The potassium-activated systems exhibited a different kinetic evolution compared with the sodium-based mixtures. Although both mixtures showed rapid thermal development during the first hours, the reaction profiles were broader and more sustained over time. In addition, the P2.0-5 mixture exhibited a characteristic double-peak behaviour during the first hours of reaction (Figure 2b). This feature may indicate the occurrence of overlapping reaction processes, possibly associated with rapid initial precipitation followed by early structural reorganisation of the reaction products.

In addition, the evolution of early-age compressive strength followed a similar trend to that observed in the calorimetric response, where mixtures exhibiting higher and faster thermal peaks also developed higher initial mechanical strengths. This correlation suggests that the rapid precipitation processes occurring immediately after activation play a key role in the initial formation of the binding structure.

Overall, the calorimetric results indicate that the investigated systems are governed by highly dynamic early-age reactions, strongly influenced by activator chemistry and concentration [32], [33]. The rapid initial thermal evolution supports the interpretation that precipitation processes dominate the earliest reaction stages, whereas the later evolution appears to be controlled by progressive reorganisation of the gel structure [34]. These observations are consistent with the FTIR results discussed in Section 3.6, where the progressive shift of the Si–O–T band suggests continuous microstructural refinement with curing time.

3.3 Mechanical performance

Figure 3(a) shows the evolution of compressive strength with curing time for the studied mixtures. All mixtures exhibited a progressive increase in strength, although clear differences were observed in both early-age development and long-term performance. Among the sodium-based systems, S2.5-6 showed the highest early-age strength, reaching 12.2 MPa at 3 days and 14.4 MPa at 7 days. This indicates a faster initial reaction and a more rapid formation of binding phases during the first stages of curing. However, its strength gain slowed down at later ages, reaching approximately 21.5 MPa at 120 days.

In contrast, S1.5-5 and S1.85-5 developed strength more slowly at early ages but showed the highest long-term performance. At 120 days, these mixtures reached 31.5 MPa and 29.1 MPa, respectively, indicating that a slower initial reaction did not limit their final mechanical development. This behaviour suggests that the long-term evolution of the binding matrix was more favourable in these mixtures, probably due to a progressive refinement and densification of the reaction products with curing time.

The potassium-based mixtures showed lower early-age strength compared with the most reactive sodium-based system. P1.85-6 and P2.0-5 reached only 3.6 MPa and 2.9 MPa at 3 days, respectively. Nevertheless, both mixtures continued gaining strength over time, reaching 21.7 MPa and 20.1 MPa at 120 days. This delayed strength development suggests a slower reaction process, but also confirms that these systems were able to develop a mechanically stable matrix at later ages.

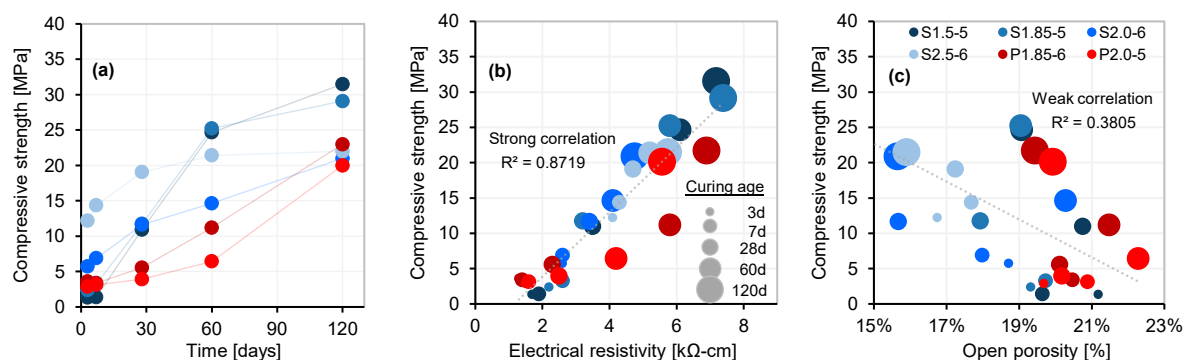


Figure 3. Mechanical performance and correlation with transport and pore structure indicators: (a) evolution of compressive strength with curing time, (b) relationship between compressive strength and electrical resistivity, and (c) relationship between compressive strength and open porosity.

Figure 3(b) relates compressive strength to electrical resistivity. A strong positive correlation was observed between both parameters, with an R^2 value of 0.8719. This indicates that the increase in mechanical strength was closely associated with the development of a less conductive and more refined pore network. Therefore, the strength gain cannot be interpreted only as a consequence of age-dependent reaction progress, but also as the result of microstructural changes that reduce ionic mobility within the hardened matrix [35].

Overall, the results show that the mixtures followed different mechanical development routes. S2.5-6 was characterised by rapid early strength development, whereas S1.5-5 and S1.85-5 achieved superior long-term performance. The strong relationship between compressive strength and resistivity supports the idea that mechanical performance is strongly linked to the progressive refinement and connectivity reduction of the pore structure. This point will be further discussed in relation to porosity, dimensional stability, and microstructural evolution in the following sections.

Figure 3(c) shows the relationship between compressive strength and open porosity. In contrast to the strong correlation observed between compressive strength and electrical resistivity, only a weak relationship was found between strength and open porosity, with an R^2 value of 0.3805. This indicates that the mechanical performance of the studied mixtures cannot be explained solely by the total volume of accessible pores. Instead, the results suggest that pore connectivity, tortuosity, and the refinement of the binding matrix play a more relevant role in strength development than open porosity alone.

3.4 Electrical resistivity and pore solution chemistry

Figure 4 presents the evolution of electrical resistivity, pH, and their relationship with porosity for the studied mixtures. A continuous increase in electrical resistivity with curing time was observed in all systems (Figure 4a), indicating a progressive reduction in ionic transport through the pore network. This behaviour reflects the ongoing formation and reorganisation of reaction products, which progressively hinder ion mobility within the hardened matrix [35].

Despite the marked increase in electrical resistivity, only relatively limited differences in open porosity were observed among the mixtures. The weak correlation between both parameters ($R^2 \approx 0.38$) indicates that ionic transport behaviour cannot be explained solely by the total volume of accessible pores. Since the reported porosity corresponds to water-accessible open porosity, it does not fully capture pore connectivity, tortuosity or pore network refinement, which appear to play a more relevant role in controlling ionic transport.

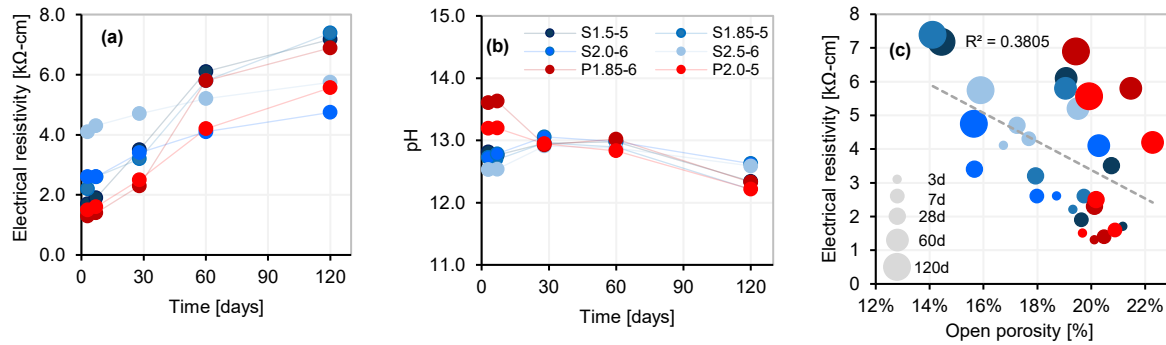


Figure 4. Electrical resistivity in relation to open porosity and pore solution chemistry: (a) evolution of electrical resistivity with curing time, (b) evolution of pH, and (c) relationship between electrical resistivity and open porosity.

This behaviour contrasts with that typically observed in conventional Portland cement systems, where a reduction in total porosity is commonly associated with improved mechanical performance and reduced ionic transport capacity. In the present study, the results suggest that the internal structure and connectivity of the pore network play a more dominant role than total open porosity alone [35], [36].

The evolution of pH with curing time (Figure 4b) shows a slight decrease for all mixtures, although the alkaline environment remains relatively stable throughout the studied period. This indicates that the pore solution maintains sufficient alkalinity to ensure the stability of the binding phases. The relatively small variation in pH further suggests that changes in ionic transport properties are not solely governed by variations in pore solution chemistry, but rather by structural modifications within the solid matrix.

Overall, the combined analysis of resistivity, open porosity, and pH indicates that ionic transport behaviour in these systems is governed by microstructural refinement and gel evolution. These results are consistent with the strong relationship previously identified between electrical resistivity and mechanical performance and provide a basis for the microstructural interpretation presented in the section 3.6.

3.5 Dimensional stability and mass loss

Figure 5 presents the evolution of drying shrinkage with curing time and its relationship with mass loss for all studied mixtures. As shown in Figure 5a, drying shrinkage increases rapidly at early ages, particularly within the first 28 days, followed by a stabilisation phase with only minor changes up to 120 days. This behaviour indicates that most of the shrinkage occurs during the initial drying period, coinciding with the early stages of microstructural development (section 3.6).

Despite the similar evolution trends observed for all mixtures, noticeable differences in shrinkage magnitude can be identified. These differences become particularly evident when shrinkage is analysed in relation to mass loss (Figure 5b). While all mixtures exhibit comparable levels of mass loss, especially at later ages, significantly different shrinkage values are observed for similar ranges of water loss.

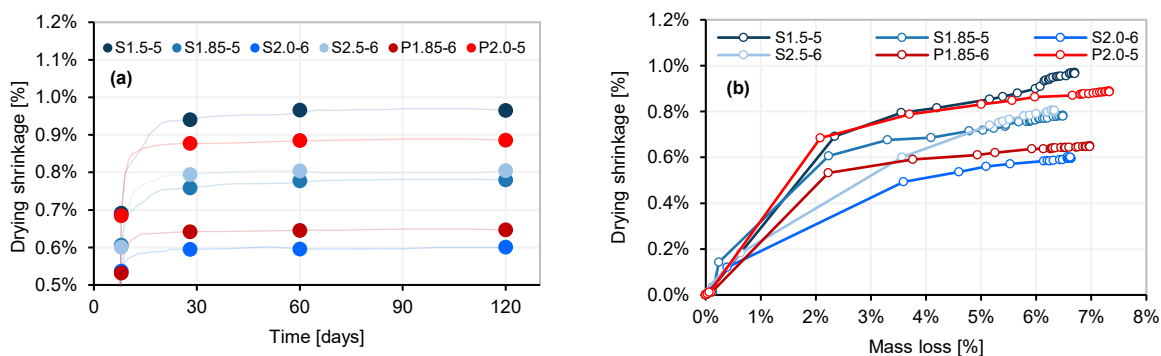


Figure 5. (a) Evolution of drying shrinkage with curing time. (b) Relationship between mass loss and drying shrinkage for all mixtures.

This behaviour indicates that drying shrinkage in these systems is not solely governed by the amount of evaporated water. It should be noted that mass loss reflects the total amount of water removed from the system, but does not provide information about the location, binding state, or transport pathways of this water within the microstructure. As a result, similar mass loss values can correspond to different internal conditions, leading to distinct shrinkage responses [37].

The observed differences in shrinkage are therefore more closely related to microstructural characteristics, such as pore size distribution, network tortuosity, and the nature of the binding gel. A more refined and connected gel structure can generate higher capillary stresses during drying, even when the total amount of water loss is comparable [35]. This interpretation is consistent with the transport results discussed in Section 3.4, where electrical resistivity was shown to be strongly influenced by microstructural refinement rather than by total open porosity alone.

Furthermore, the shrinkage behaviour aligns with the FTIR results presented in Section 3.6, where a progressive reorganisation of the gel structure was identified. The development of a more polymerised and homogeneous network can increase the sensitivity of the material to internal stresses generated during drying, contributing to the observed differences in shrinkage.

Overall, the combined analysis of drying shrinkage and mass loss highlights that dimensional stability in these systems is controlled by microstructural evolution rather than by simple water loss [38], [39]. These findings reinforce the need to consider both ionic transport and microstructural aspects when evaluating the long-term performance of alkali-activated materials.

3.6 Microstructural evolution based on FTIR analysis

Figure 6 presents the FTIR spectra of all mixtures at different curing ages (3, 28, and 120 days), together with the evolution of the position of the main Si–O–T band within the 800–1200 cm^{-1} region. All systems exhibit a broad band associated with the asymmetric stretching vibrations of Si–O–T (T = Si, Al), characteristic of the formation of aluminosilicate-type binding gels [37], [38], [39]. A progressive shift of this band towards lower wavenumbers with curing time is observed for all mixtures, with a total displacement of approximately 15–30 cm^{-1} . This shift reflects changes in the local Si–O–T bonding environment and is commonly associated with the progressive incorporation of Al- and Ca-bearing species into the reaction products [37]. In these systems, the formation of C–(N)–A–S–H-type gels leads to a modification of the Si–O–

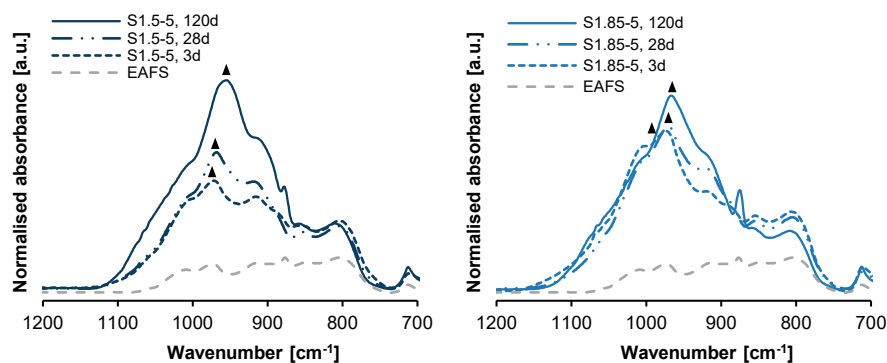
T bond environment, which may contribute to the lower vibrational frequencies observed as the reaction products become more chemically and structurally complex.

In addition to the band shift, a gradual narrowing and reduction in asymmetry of the main band can be observed with curing time, indicating a progressive reorganisation and homogenisation of the gel structure. These changes are consistent with the increase in electrical resistivity discussed in Section 3.4, as a more reorganised and refined gel network reduces pore connectivity and limits ionic transport, even in mixtures with comparable open porosity values.

Some mixtures exhibit a slightly different behaviour at intermediate ages (28 days), where the band position appears temporarily shifted towards higher wavenumbers. This behaviour may be associated with a transient stage of the reaction, in which the dissolution of slag phases is not yet complete and the incorporation of Al- and Ca-bearing species into the gel is still limited. As curing progresses, the continued release of these species promotes further structural reorganisation, resulting in the shift towards lower wavenumbers observed at 120 days. This interpretation is consistent with the mechanical behaviour described in Section 3.3, where certain mixtures show slower early development but improved long-term performance.

It should be noted that the band position reported in this study corresponds to an approximate location of the main Si–O–T band within the analysed region, rather than a strictly defined peak maximum. This approach is appropriate given the broad and overlapping nature of the bands in these materials.

To further support the interpretation of the Si–O–T band evolution, a semi-quantitative deconvolution of the 700–1200 cm^{-1} region was performed on selected representative mixtures (Figure 6). Prior to fitting, the spectra were baseline-corrected and area-normalised, allowing the relative contribution of the fitted components to be compared between curing ages. Gaussian functions were used, and the minimum number of stable components required to reproduce the spectral envelope was selected in each case, avoiding residual, highly dependent or poorly defined components introduced solely to improve the goodness of fit.



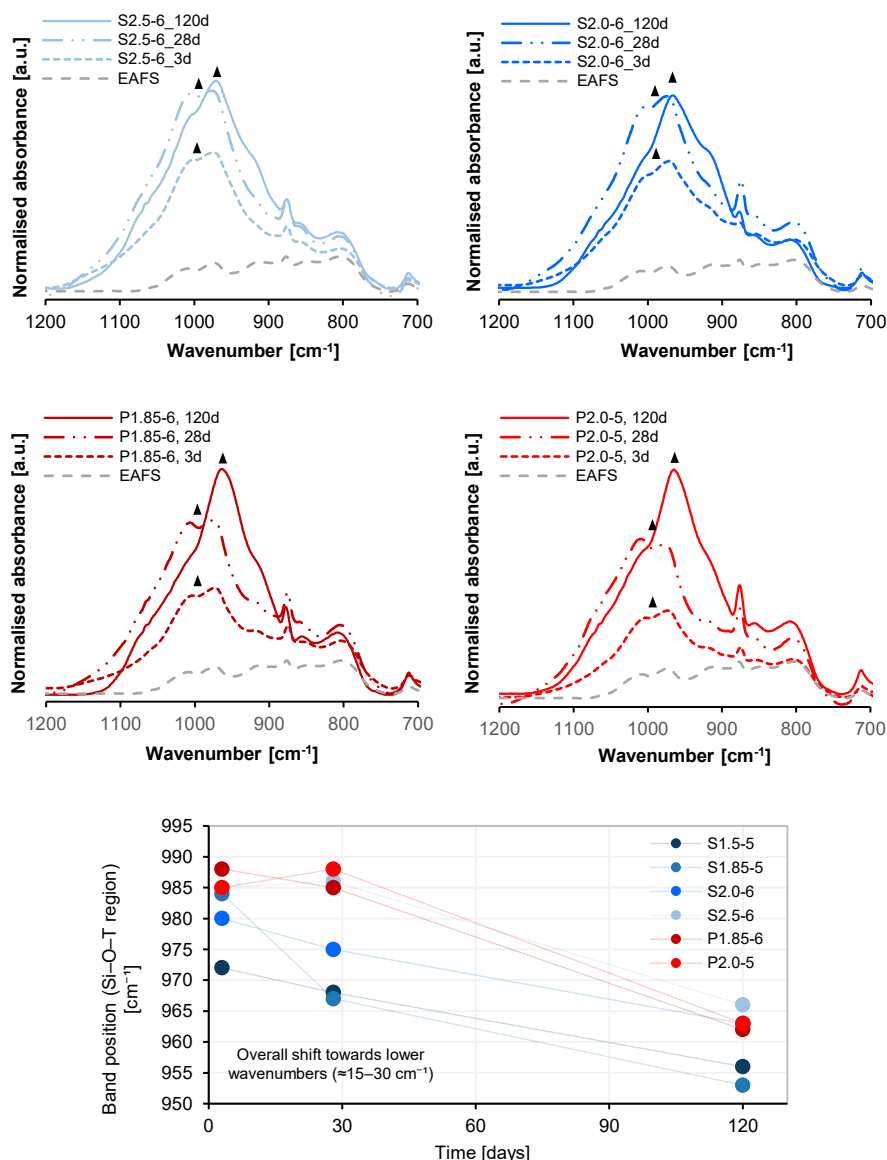


Figure 6. FTIR spectra of the studied mixtures at 3, 28, and 120 days of curing, showing the evolution of the main Si–O–T (T = Si, Al) band in the 800–1200 cm⁻¹ region.

For comparative purposes, the fitted components were assigned to approximate spectral regions according to their centre position, rather than being interpreted as individual Qⁿ species. Four regions were considered: R1, low-frequency region, 700–830 cm⁻¹; R2, intermediate region, 830–930 cm⁻¹; R3, main Si–O–T region, 930–1000 cm⁻¹; and R4, high-frequency region, 1000–1200 cm⁻¹. This regional classification was used only to describe the redistribution of the Si–O–T band during curing. It should not be regarded as a direct quantification of specific Qⁿ units, given the strong overlap of the FTIR bands and the possible contribution of residual precursor phases and Fe-bearing environments.

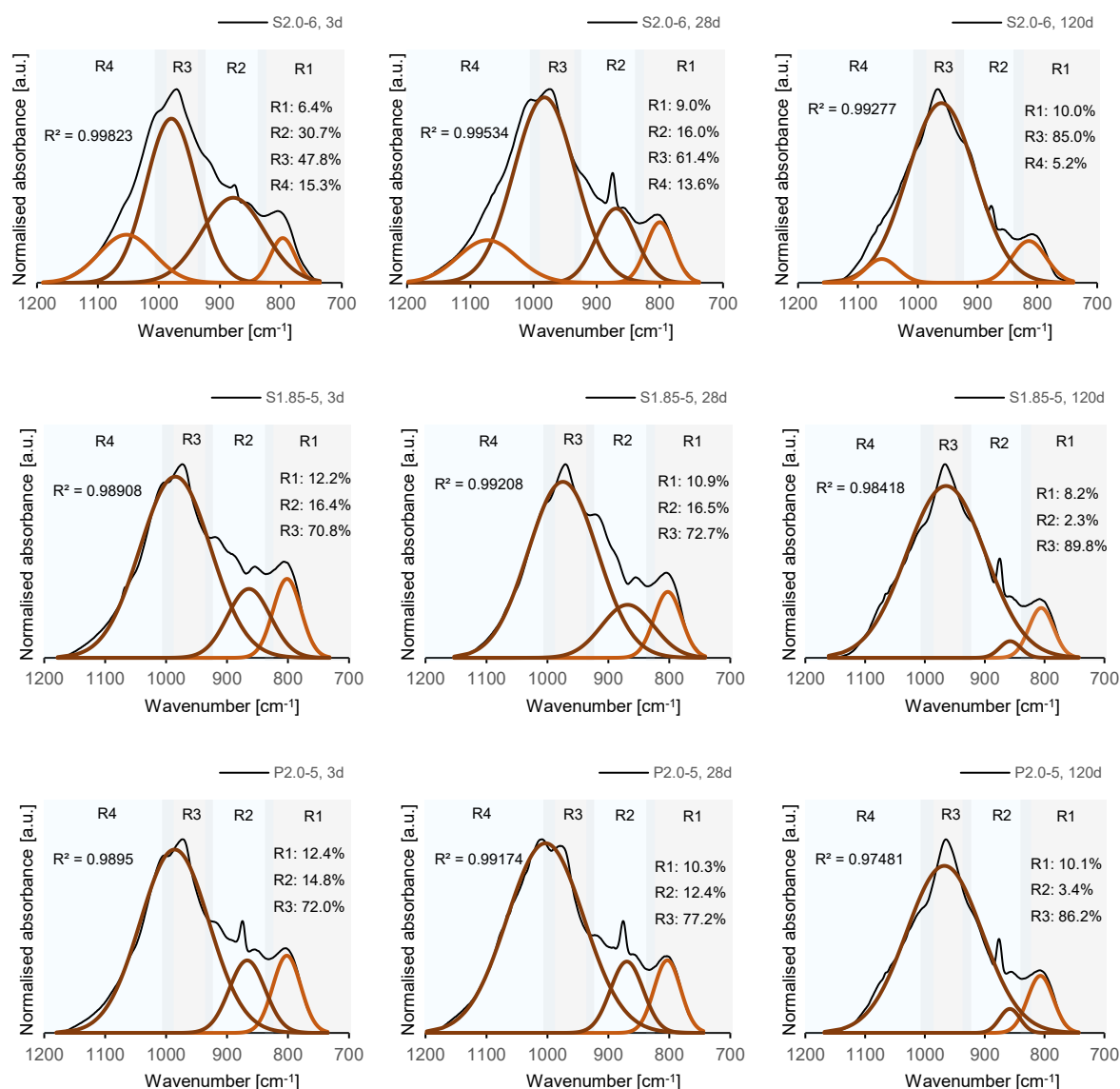


Figure 7. Representative deconvolution of the Si–O–T band in selected sodium- and potassium-activated EAFS mixtures at 3, 28, and 120 days of curing. The fitted components are grouped according to the R1–R4 spectral regions defined in the text, and the percentages indicate their relative area contributions after area normalisation.

The deconvolution results provide a complementary view of the spectral evolution already observed from the displacement and narrowing of the main Si–O–T band. In S2.0-6, representative of the sodium-activated mixtures with higher silica modulus, the contribution of the main Si–O–T region, R3, increased from 47.8% at 3 days to 61.4% at 28 days and 85.0% at 120 days. At the same time, the intermediate region, R2, decreased and was no longer clearly resolved at 120 days. This behaviour indicates a progressive simplification of the spectral envelope and a greater concentration of the signal in the main Si–O–T region.

A similar tendency was observed for S1.85-5, representative of the sodium-activated mixtures with lower silica modulus. In this case, R3 remained the dominant contribution throughout curing, increasing from 70.8% at 3 days to 72.7% at 28 days and 89.8% at 120 days. The progressive reduction of the low- and intermediate-frequency contributions is consistent with

the sharpening of the band observed in the original FTIR spectra, suggesting a gradual homogenisation of the Si–O–T environments.

In the potassium-activated mixture P2.0-5, R3 was also the dominant contribution, increasing from 72.0% at 3 days to 77.2% at 28 days and 86.2% at 120 days. Although the position of the main band did not evolve monotonically in all potassium-activated systems, the deconvolution indicates a progressive concentration of the spectral contribution in the main Si–O–T region. This supports the interpretation that the gel structure continues to reorganise during curing, in agreement with the delayed strength development and the progressive increase in electrical resistivity observed for these mixtures.

Overall, the deconvolution results show that the evolution of the Si–O–T band involves not only a shift in band position, but also an internal redistribution of the relative spectral contributions. The progressive predominance of the main Si–O–T region, together with the reduction or disappearance of intermediate contributions at later ages, is consistent with the gradual reorganisation and homogenisation of the binding gel. However, due to the overlapping nature of the FTIR bands and the chemical complexity of Fe-rich EAFS systems, these results should be interpreted as indirect evidence of changes in silicate environments, rather than as direct structural identification of specific Q^n species.

Considering the chemical composition of the precursor, the potential role of Fe should also be taken into account. Electric arc furnace slag typically contains significant amounts of Fe, predominantly in the form of Fe^{2+} within the original structure. Previous studies on Fe-rich alkali-activated systems have shown that, upon activation, partial oxidation to Fe^{3+} may occur, and that Fe^{3+} can participate in the formation of reaction products. In particular, Fe^{3+} has been proposed to be incorporated into the aluminosilicate network in tetrahedral coordination, in a manner analogous to Al^{3+} , whereas Fe^{2+} is more likely to remain in octahedral environments or in Fe-rich secondary phases.

In this context, the observed evolution of the Si–O–T band may not be exclusively related to the incorporation of Al and Ca, but could also be influenced by the presence and participation of Fe-bearing species within the binding structure [4], [40]. However, it must be emphasised that FTIR does not allow distinguishing between Si–O–Al and Si–O–Fe linkages, nor does it provide direct information on the oxidation state or coordination environment of Fe. Therefore, the involvement of Fe in the gel structure is considered here as a plausible mechanism supported by previous studies, rather than as direct evidence derived from the present results.

Overall, the FTIR analysis supports the interpretation that the microstructural evolution of the studied systems is governed by progressive gel formation and structural reorganisation, rather than by simple changes in pore volume. This refinement of the internal structure provides a consistent explanation for the increase in electrical resistivity and mechanical strength observed in previous sections, reinforcing the idea that the performance of these materials is primarily controlled by the connectivity and organisation of the gel network.

3.7 Mechanisms governing material behaviour

The combined results obtained in this study indicate that the behaviour of the investigated systems is primarily governed by microstructural evolution, rather than by simple changes in total porosity or mass loss. By integrating the mechanical, transport, FTIR, and dimensional stability results, a consistent interpretation of the governing mechanisms can be established.

At early ages, the reaction appears to be characterised by rapid precipitation of reaction products following the initial dissolution of reactive species. This leads to the formation of a relatively

heterogeneous and weakly connected gel structure. As reflected in the mechanical performance (Section 3.2) and the FTIR results (Section 3.5), this early-stage gel exhibits a less developed and less reorganised network. Consequently, the pore structure remains relatively open, which is consistent with the lower electrical resistivity values observed in Section 3.3.

With increasing curing time, the gradual release of reactive species promotes the incorporation of Al- and Ca-bearing species into the binding gel, leading to progressive structural reorganisation. This evolution is supported by the shift of the Si–O–T band towards lower wavenumbers in the FTIR analysis, which suggests changes in the local Si–O–T bonding environment and progressive gel reorganisation. As the structure becomes more refined, pore connectivity is reduced and tortuosity increases, leading to a marked increase in electrical resistivity, even in systems with comparable open porosity.

The potential contribution of Fe-bearing species should also be considered. The precursor contains significant amounts of Fe, predominantly in the form of Fe^{2+} , which may partially oxidise to Fe^{3+} during activation. Previous studies have suggested that Fe^{3+} can participate in the formation of binding structures, either by incorporation into the aluminosilicate network or through the formation of Fe-rich gel domains [14]. Although this contribution cannot be directly confirmed by FTIR, it may influence the structural evolution of the gel and, consequently, the transport behaviour of the material.

The dimensional stability results further support this interpretation. Despite similar levels of mass loss among mixtures, significantly different shrinkage values were observed. This indicates that drying shrinkage is not governed solely by the amount of water removed, but rather by the internal structure of the pore network [41], [42]. In more refined systems, the presence of smaller and more connected capillary pores can lead to higher internal stresses during drying, resulting in increased shrinkage.

Overall, the results consistently show that mechanical performance, transport properties, and dimensional stability are controlled by the evolution of the gel structure and the associated pore network characteristics. This conceptual framework is schematically illustrated in Figure 8. These results confirm that, in alkali-activated systems, total porosity and mass loss alone are not sufficient descriptors of material behaviour, and that microstructural organisation plays a dominant role in defining performance [41].

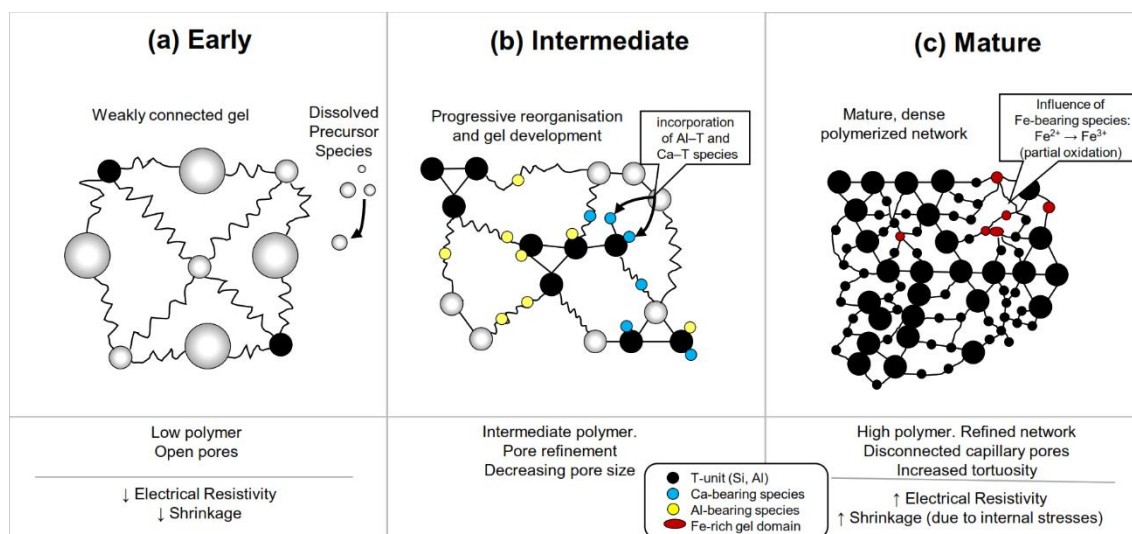


Figure 8. Schematic representation of the mechanisms governing material behaviour, illustrating the evolution of gel structure and pore network and its influence on ionic transport properties and drying shrinkage.

4 Conclusions

The findings of this study highlight the complex interaction between activator chemistry, reaction kinetics, microstructural development and macroscopic performance in EAFS-based alkali-activated systems. Rather than being controlled only by total porosity or water loss, the mechanical, ionic transport and dimensional stability responses were mainly governed by the progressive organisation of the binding gel and the refinement of the pore network. Based on this integrated interpretation, the following conclusions can be drawn.

1. The alkali activation of EAFS strongly depended on the chemistry and dosage of the activating solution. Mixtures with higher alkali content and/or higher silica modulus, particularly S2.0-6 and S2.5-6, exhibited a more intense kinetic response and superior mechanical performance at early ages. However, the behaviour was not governed solely by the type of alkaline cation, since the potassium-activated mixtures exhibited relatively short setting times and higher early-age strengths than some sodium-based formulations with lower reactivity.
2. Semi-adiabatic calorimetry showed that the early-age behaviour of the investigated systems was dominated by rapid precipitation reactions immediately after activation. The calorimetric response followed trends consistent with the development of early-age compressive strength, suggesting that the rapid formation of initial reaction products plays a key role in the formation of the binding structure.
3. FTIR analyses revealed a progressive shift of the main Si–O–T band towards lower wavenumbers with curing time, indicating changes in the Si–O–T bonding environment and progressive structural reorganisation of the gel network. These results suggest that the microstructure of the investigated systems continues evolving well beyond the early curing stages.
4. The results demonstrated that ionic transport properties and dimensional stability were not governed solely by total open porosity or drying mass loss. Mixtures exhibiting similar porosity and comparable mass loss developed significantly different electrical resistivity and shrinkage behaviour, highlighting the dominant role of pore network organisation, connectivity, and tortuosity.
5. Considering the high iron content of the precursor, the possible participation of Fe species in the structural evolution of the gel was discussed. Although the techniques employed do not allow direct confirmation of Fe incorporation within the binding structure, the results suggest that iron may influence the long-term microstructural evolution and transport behaviour of the investigated systems.
6. The integrated interpretation of calorimetry, FTIR, mechanical performance, ionic transport properties, and dimensional stability demonstrated that the behaviour of alkali-activated EAFS systems was primarily governed by gel evolution and pore network refinement rather than by simple variations in total porosity. These findings contribute to improving the understanding of Fe-rich alkali-activated systems and support the valorisation of low-commercial-value fine EAFS fractions as sustainable precursors for construction materials.

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Data availability

The datasets generated and/or analysed during the current study are available from the corresponding author upon reasonable request.

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Author contributions

M.R.-R. designed the experimental programme, carried out the experimental work, analysed the data, prepared the figures and wrote the original draft. D.A. contributed to the conceptualisation of the study, data interpretation, resources, project management, supervision and manuscript revision. M.B.-B. contributed to the conceptualisation of the study, data interpretation, resources, project management, supervision and manuscript revision. All authors reviewed and approved the final manuscript.

Additional information

Competing interests

The authors declare no competing interests.