



Impact of curing parameters on the properties of geopolymers derived from different class F fly ashes

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Abstract

This study investigates the effect of curing temperature and duration on the properties of Class F fly ash-based geopolymers (FGPs). FGP samples produced from three different fly ash sources were cured under various temperatures (ambient, 60 °C, 75 °C, and 90 °C) and durations (8, 24, 72, and 168 h). The compressive strength development, phase characterisation (FTIR and XRD) and microstructural evaluations (SEM and MIP) were performed. The major results reveal that although the chemical compositions of the fly ashes are quite similar, properties such as particle size distribution and particle morphology have a substantial impact on the properties of the FGPs. Fly ashes with a particle size distribution in a narrow range and mostly spherical morphology show higher reactivity, thereby minimising the requirement for extreme curing conditions. Therefore, FGPs produced with high-reactivity fly ash achieved high strength even at lower curing temperatures and shorter durations.

Keywords Fly ash · Particle size distribution · Geopolymer · Compressive strength · Phase characterisation

Introduction

The need for concrete as a construction material is gradually increasing [1]. The production of Ordinary Portland Cement (OPC), which is a constituent of concrete, is also increasing in parallel with the demand for concrete. Global production of OPC has approached 4 billion tonnes annually [2]. However, serious environmental concerns emerge during this production, especially due to the greenhouse gas emissions [3]. For example, for every tonne of OPC produced, approximately 0.7–1.1 tonnes of carbon dioxide (CO₂) is released into the atmosphere, causing global warming [4–8]. Further, the OPC production causes nitric oxide emission, leading to acid rains [4]. Therefore, the development of new binders as

alternatives to OPC is critical to maximise environmental benefits [9]. In recent years, geopolymers have been developed as promising alternatives to OPC, primarily because of their eco-friendly characteristics [10]. They also present enhanced mechanical properties and stability in volume [4], show high resistance to acid attacks and elevated temperatures [1], and represent high early strength [11].

Geopolymers are generally characterised as amorphous aluminosilicate cementitious materials [12]. They are formed through the reaction of aluminosilicate materials with alkali activators [9]. Geopolymerisation is a polymeric reaction that relies on the alumino-silica chain, occurring when a specific quantity of alumina and silica reacts in an alkaline solution. This process is commonly termed as alkali activation, a process that transforms the amorphous components of materials into a composite with strong binding properties [11]. Even though studies are being conducted to fully understand all the processes of alkali activation with aluminosilicates, the most widely accepted process for geopolymerisation can be summarised in three steps [4, 13]: (i) the dissolution of amorphous aluminosilicate materials at high pH, (ii) transportation of precursor ions into monomers, and (iii) condensation of monomers into polymers and hardening into geopolymer structure.

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To produce geopolymers, a wide range of naturally occurring and industrially produced aluminosilicate materials can be used as precursors [9, 14]. Among these, the most commonly used precursors are calcined clays, ground granulated blast furnace slag, and fly ash. However, calcined clays are less preferable because of their plate-shaped particles, leading to high water demand and porosity in concrete production [15]. In addition, as a hazardous waste, the need to recycle slag is significantly lower compared to fly ash [16]. Therefore, fly ash, known as industrial by-product, is a more suitable precursor for the geopolymerisation [1].

Fly ash is a by-product produced in thermal power plants through the combustion of coal [17, 18]. In many countries, fly ash is generated in large amounts, with over 65% of this by-product currently being disposed of in landfills [19]. John et al. [9] indicated a global annual production of approximately 780 million tonnes of fly ash, with only about 17–20% being effectively utilised. This inadequate management of fly ash as a waste material poses potential risks of severe environmental and health issues [20]. Moreover, fly ash production continues to increase in countries such as China, India, and Turkey where coal-fired power plants are dominantly used for electricity generation [16].

Curing conditions represent one of the critical parameters influencing the geopolymerisation process [2]. In an alkaline environment, the dissolution rates of binders such as slag and metakaolin are high [21, 22], class F fly ash dissolves at a moderate rate [11, 23], and natural pozzolans dissolve at a slower rate [24, 25]. To this direction, the need for curing temperature and duration increases in relation to the rate of dissolution. The geopolymerisation process of a fly ash-based geopolymer (FGP) is quite slow at ambient temperature, whereas the rate of the reaction process accelerates with an elevated temperature [11, 26].

Table 1 provides an overview of previous studies on how curing temperature, curing duration and testing age affect FGPs, and also lists the curing conditions that those studies considered to be optimal. In several studies, strength increases were observed at temperatures or durations higher than the recommended values, and some included partial replacement of fly ash, which may have affected the reported outcomes. For this reason, it is difficult to define a single curing regime that is applicable in all cases. Nevertheless, most studies indicate that low-calcium fly ashes generally perform best within a curing range of about 60–90 °C. Variations within this range are mainly associated with differences in activator composition and fly ash chemistry. However, even fly ashes with similar chemical compositions may require different curing conditions due to differences in particle size, morphology, surface area, amorphous content and dissolution behaviour.

Most earlier literature has concentrated on the chemical composition of individual low-calcium fly ashes, while the roles of their physical and microstructural characteristics have received far less attention. This study addresses this gap by examining three low-calcium fly ashes from the Catalagzi, Iskenderun and Sugozy power plants, which show similar chemical compositions but differ in several key properties. These materials were characterised using specific surface area measurements, Blaine and BET analyses, scanning electron microscopy (SEM), X-ray diffraction (XRD) and nitrogen adsorption. The first aim of the study is to investigate how these characteristics influence the compressive strength of FGPs, rather than relying solely on chemical composition. Compressive strength was assessed at 28 days for all sources, and extended to 3, 7 and 90 days for Catalagzi fly ash, which is unique in Türkiye for its use of domestically mined bituminous coal.

Although many studies have proposed curing ranges for improving FGP strength, the most suitable combination of curing temperature and duration remains uncertain. Therefore, the second aim of this work is to examine how these two parameters affect the mechanical and microstructural development of Catalagzi FGPs synthesised with sodium silicate and sodium hydroxide solutions. Samples were cured at ambient temperature, 60 °C, 75 °C and 90 °C for 8, 24, 72 and 168 h. Compressive strength testing was supported by Fourier transform infrared spectroscopy (FTIR), XRD, SEM and mercury intrusion porosimetry (MIP) analyses to provide insight into microstructural evolution and pore characteristics.

Experimental programme

Materials

Three different Class F fly ashes were used to produce FGPs. The first one was obtained from Catalagzi Thermal Power Plant (Zonguldak, Türkiye), the only power plant that uses domestic bituminous coal as fuel in the country. The second and third fly ashes were obtained from Iskenderun Thermal Power Plant (Hatay, Türkiye) and Sugozy Thermal Power Plant (Adana, Türkiye), respectively. The fly ashes used in this study were sourced from Turkish thermal power plants employing typical pulverised coal combustion technologies; fluidised bed combustion systems were not considered within the scope of this study. The locations where these three fly ashes were obtained are shown with the regional and global scales in Fig. 1.

The chemical compositions determined by wet-chemical analysis of fly ashes are presented in Table 2. It is evident that the contents of CaO, SiO₂, Al₂O₃, and Fe₂O₃, which are

Table 1 Literature review on curing temperatures, durations, and testing age for class F fly ash-based geopolymers

References	Curing temperature range (°C)	Curing time range (h)	Optimum curing condition	Testing age (days)
Palomo et al. 1999 [27]	65 and 85	2, 5, and 24	85 °C for 5/24 h	Single age (after curing)
Swanepoel and Strydom 2002 [28]	40, 50, 60, and 70	6, 24, 48, and 72	60 °C for 48 h	7 and 28
Criado et al. 2005 [29]	85	5, 12, 20, and 168	-	Single age (unspecified)
Hardjito and Rangan 2005 [30]	30, 45, 60, 75, and 90	4, 6, 8, 12, 24, 48, 72, and 96	-	3, 7, 14, 28, 56, and 91 (only for specific curing conditions)
Sindhunata et al. 2006 [31]	30, 50, and 75	24 and 48	-	No strength results.
Vora and Dave 2013 [32]	60, 75, and 90	24, and 48	Studied separately.	3 and 7 (only for specific curing conditions)
Adam and Horianto 2014 [33]	Ambient, 80, 100, and 120	4, 6, and 20	120 °C for 20 h	3, 7, 14, and 28
Atiş et al. 2015 [11]	45, 55, 65, 75, 85, 95, 105, and 115	24, 48, and 72	115 °C for 24 h	1 for 24 curing time; 2 for 48 curing time; 3 for 72 curing time
Nagalia et al. 2016 [34]	Oven-curing: 55 and 70 °C; Steam-curing: 46 °C; Ambient-curing: 25 °C	24, 72, and 168	70 °C for 48 h	1, 7, and 28 (Only for specific curing conditions)
Görhan et al. 2016 [21]	60 and 80	2, 4, and 24	60 °C for 2 h	7
Aliabdo et al. 2016 [35]	28, 50, 70, and 90	24, 48, and 72	70 °C for 48 h	7 and 28
Zhang et al. 2018 [36]	At ambient temperature, at 50 °C for 168 h, and at 80 °C for 24 h	-	-	2, 7, 14, 28, 49, 90, and 120
Hassan et al. 2019 [37]	Ambient, 75	26	75 °C for 26 h	7, 28, 56, 90, and 180
Kuenzel and Ranjbar 2019 [38]	25, 45, 65, 85, 105, 125, 145	1, 2, 4, 6, 12, and 24	-	No strength results.
Sajan et al. 2021 [39]	20, 40, 60, and 80	72, 168, and 336	60 °C for 168 h	14
Rabie et al. 2022 [40]	Ambient, 40, 80, and 120	24, 48, and 72	80 °C for 24 h	1, 3, 7, and 28 (only for ambient and 80 °C for 24 h)
Gopalakrishna and Dinakar 2023 [2]	Ambient, 60, 80, and 100	24	80 °C for 24 h	3, 7, 28, 56, and 90
Yılmaz et al. 2023 [23]	Ambient, 40, 60, and 80	24, 48, and 72	80 °C for 72 h	2, 7, and 28
Karaaslan et al. 2025 [25]	Ambient (25 °C), high-ambient (45 °C), and heat-curing (at 75 °C for 24 h)	-	High-ambient curing	3, 7, 28, and 90

[29] Strength continued to increase with curing time, reaching higher values up to 168 h

[30] The full range of curing temperature and duration combinations was not investigated. Increasing temperature enhances strength, but improvement is limited above 60 °C. At 60 °C, longer curing time increases strength noticeably up to 24 h

[21] Metakaolin was used as a replacement for fly ash at 10%, 20%, 30%, and 40%

[35] OPC was used as a replacement for fly ash at 5%, 10%, and 15%. Also, curing at 70 °C for 72 h was not tested

[36] Red mud was used as a replacement for 25% fly ash

[39] Curing at 60 °C for 168 h was optimal, as 80 °C and 336 h offered no significant strength gain

critical for geopolymerisation in powder binders, are largely comparable across the three fly ashes.

In order to activate the fly ashes, a mixture consisting of commercial-grade sodium hydroxide (with 98% purity) and sodium silicate (with a weight ratio of $\text{SiO}_2/\text{Na}_2\text{O}=2.04$ and a SiO_2 content of 24.50%) was used. The sodium hydroxide was dissolved in distilled water to obtain a 10 M NaOH solution, which was then left to rest for 24 h before use. Subsequently, it was added to the sodium silicate solution, and after 5 min of stirring, the mixture was allowed to stand for half an hour. After this period, finally, the mixture was added to the solid material consisting of fly ash and sand.

In addition, sand (with a maximum particle size of 4.75 mm) obtained from the local river deposits was utilised as aggregate. This sand has a specific gravity of 2.71 and a

water absorption rate of 1.6%. The sand used in this study was graded in accordance with ASTM C144-04 [41].

Sample preparation and curing

In terms of samples, three different FGP mortar mixtures were prepared with three different fly ashes. Based on the same consistency, the mixing ratios were selected as shown in Table 3. Also, the weight ratio of sodium silicate solution to 10 M NaOH solution used in these mortars was 2.5 for all mixtures. The weight ratio of the alkali activator solution to fly ash was 0.50 for Catalagzi and Iskenderun, and 0.35 for Sugozi.

The mixing process for all mixes was conducted in the same way. The fly ash and sand were firstly mixed in

Fig. 1 Locations of thermal power plants where the fly ashes were collected, global and Türkiye regional scales

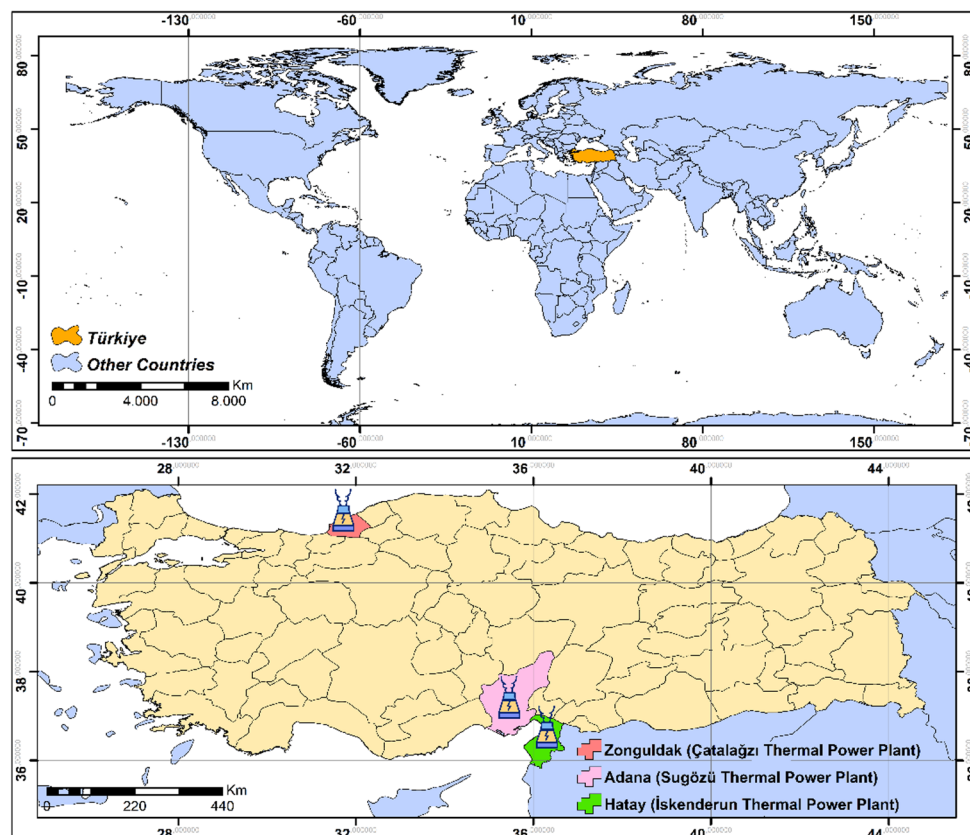


Table 2 Chemical compositions of fly ashes (wt%)

Fly ashes	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	LOI
Catalagzi	2.46	55.51	23.09	6.64	1.58	0.14	3.56	0.24	1.63
Iskenderun	3.17	58.36	25.57	6.85	1.97	0.09	1.49	0.04	1.26
Sugozu	3.75	55.98	21.19	6.94	2.45	1.91	2.42	0.42	2.14

LOI: Loss on ignition

Table 3 Mix proportions of FGP mortars (g)

Mix ID	Sodium hydroxide solution	Sodium silicate solution	Fly ash	River sand
Catalagzi-FGP	64.3	160.7	450	1350
Iskenderun-FGP	64.3	160.7	450	1350
Sugozu-FGP	45.0	112.5	450	1350

a cement mixer for 2 min. Then, the previously prepared alkali activator mixture was added to the mixture of solids, and the mixing was continued for another 3 min. After this time, the fresh mortar was poured into the 40 mm plastic cube molds in 2 layers. For each layer, the cube molds were dropped 60 times on a jolting table. Once the placement and compaction processes were completed, the samples were wrapped in a special plastic bag to protect the moisture contents of the samples. Subsequently, the samples were placed in the oven at specified temperatures as soon as their surface finishing were completed.

At the end of the curing periods, the samples were removed from the molds and left in the laboratory (at 25 °C)

until the day tests are conducted. It should be noted here that Catalagzi and Iskenderun FGP samples left at room temperature did not solidify sufficiently within the first 3 days, so their 3-day strengths could not be measured, and they were demolded after 6 days.

Experimental methods

The compressive strengths of the Catalagzi FGPs at 3, 7, 28, and 90 days, as well as the Iskenderun and Sugozu FGPs at 28 days, were determined in accordance with TS EN 196-1 [42]. The compressive strength was determined based on the average of three specimens.

The particle size distribution of the raw fly ashes was analysed using a laser diffraction particle size analyser (Malvern Mastersizer 3000).

The nitrogen adsorption technique, using a Micromeritics Tristar II 3020 analyser, was applied to the raw fly ashes to determine their Brunauer–Emmett–Teller (BET) surface areas.

To determine the reactive Si and Al content of fly ash for geopolymerisation, 7.5 g of fly ash was mixed with 200 mL of 10 M NaOH solution in a closed beaker. The beaker was heated on a hot plate set to 75 °C, and the mixture was continuously stirred for 4 h using a magnetic stirrer. Afterward, the solution was filtered, and the filtrate was analysed using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) to measure the Si and Al content [43, 44].

The Catalagzi FGP specimens used for determining the 28-day compressive strength were further analysed using FTIR, XRD, SEM, and MIP techniques. A portion of these samples, in their bulk form, was used for SEM and MIP analyses. The remaining portion was processed into a powdered state through grinding in a ring mill. The powdered materials were subsequently employed in FTIR and XRD analyses.

To observe the molecular bond structure of Catalagzi FGPs concerning curing temperature and time, the Agilent Cary 630 model FTIR spectrometer was employed. This instrument operated in the wavelength range of 4000–650 cm^{-1} .

The mineralogical composition of the Catalagzi FGPs was determined using the Tongda TD-3500 model XRD device. The samples were measured with 2θ range of 10° to 80° in the step size of 0.02°.

The microstructure of the raw fly ashes and Catalagzi FGPs was determined using a ZEIS Gemini Sigma 300 SEM.

The pore structure of the Catalagzi FGPs was analysed through mercury intrusion porosimetry (MIP) using a Quantachrome Pore Master 60 device. This method determined the pore size distribution within the range of 4 nm to 10 μm , utilising a high-pressure chamber capable of reaching up to 55,000 psi.

Results and discussions

Effects of characterisation for Catalagzi, Iskenderun, and Sugozi fly ashes

Fly ash sources usually contain some unburned carbon particles. These particles are typically larger than 100 μm , increasing the surface area due to their porous structure [45]. Beyond the presence of unburned carbon, fly ash does not consist of a uniform particle mixture. Excluding certain impurities, fly ash particles are generally classified into three main groups [38, 46]: (i) aluminosilicates with a high amorphous fraction, (ii) cenospheres, and (iii) metal oxides, such as quartz and iron oxides. Aluminosilicates are reactive particles that fully participate in geopolymerisation. These particles typically range in size from 1 μm to 100 μm , with the majority being smaller than 50 μm [47]. Cenospheres, on the other hand, are partially reactive particles with aluminosilicate shells that react in highly alkaline environments while their skeletal structure remains unreacted. These particles range from 10 μm to 500 μm , with most being smaller than 200 μm [48]. These hollow particles typically have a specific gravity of less than 0.8 [49, 50]. Metal oxide particles, however, are inert components that do not participate in geopolymerisation and remain unchanged within the matrix, typically ranging in size from 1 μm to 40 μm . In addition to these common particle types, submicron-sized particles associated with trace elements are also presented in fly ash sources [47].

Figure 2 presents the particle size distributions for the fly ashes used in this study. Table 4 presents the D_{10} , D_{50} , and D_{90} values obtained from laser particle size distribution (LPSD) analysis, along with the specific surface area. Additionally, the Blaine surface area, BET surface area, and specific gravity values are provided. D_{10} and D_{90} values are 1.75 μm and 97.90 μm for Catalagzi, 2.83 μm and 107.00 μm for Iskenderun, and 3.87 μm and 82.10 μm for Sugozi, respectively. Catalagzi fly ash contains 7.42% particles

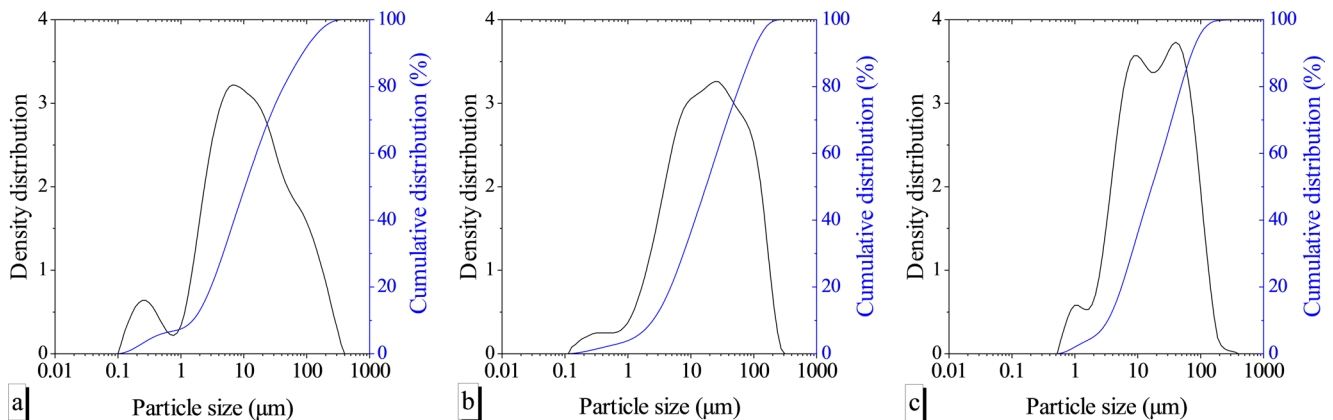
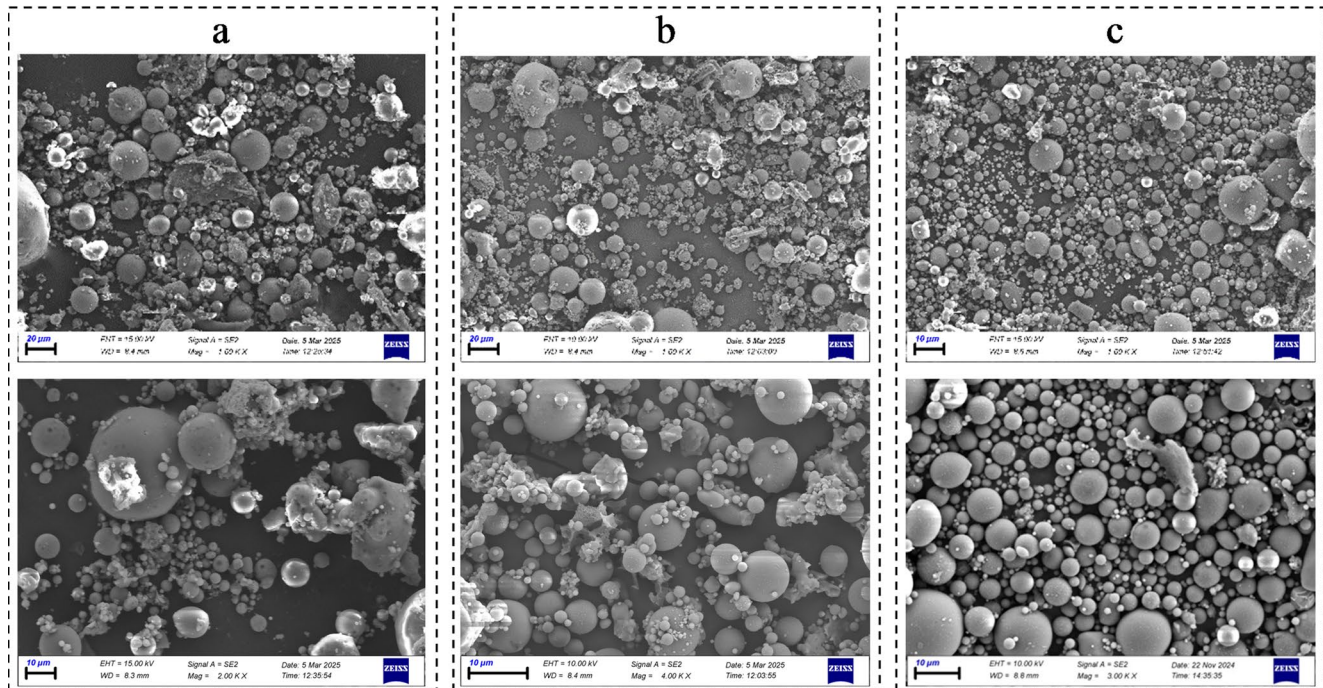


Fig. 2 Particle size distributions of Catalagzi (a), Iskenderun (b), and Sugozi (c) fly ashes

Table 4 Summary of fly ash characteristics: results of LPSD analysis, Blaine surface area, BET surface area, and specific gravity

Fly ashes	D ₁₀ (μm)	D ₅₀ (μm)	D ₉₀ (μm)	Surface area of LPSD (cm ² /g)	Blaine specific surface area (cm ² /g)	BET surface area (cm ² /g)	Specific gravity
Catalagzi	1.75	11.80	97.90	16,380	3805	19,800	2.13
Iskenderun	2.83	19.70	107.00	12,080	3295	11,300	2.12
Sugozu	3.87	19.00	82.10	7220	3004	10,850	2.34

**Fig. 3** SEM micrographs of Catalagzi (a), Iskenderun (b), and Sugozu (c) fly ashes

smaller than 1 μm and 8.38% particles larger than 100 μm. Iskenderun fly ash contains 3.91% particles smaller than 1 μm and 9.20% particles larger than 100 μm. On the other hand, Sugozu fly ash contains 2.00% particles smaller than 1 μm and 4.40% particles larger than 100 μm. Sugozu fly ash has a higher proportion of particles in the 1–100 μm range, indicating the presence of more reactive aluminosilicate particles. Moreover, the specific gravity of Sugozu fly ash is higher than that of the other two. The lower specific gravity of Catalagzi and Iskenderun fly ashes suggests that these materials may contain more low-density cenospheres compared to Sugozu fly ash [49–51].

When comparing the specific surface area, Blaine surface area, and BET surface area, Sugozu fly ash exhibits a lower surface area. This observation indicates the presence of irregular particle shapes and some unburned organic matter in Iskenderun and particularly Catalagzi fly ashes [52]. The lower surface area of Sugozu fly ash also explains why Sugozu FGP mixtures achieve similar consistency with lower alkali activator content compared to two other FGP mixtures (Table 3).

SEM images of fly ashes are shown in Fig. 3. In general, spherical particles are predominant in all samples, but Sugozu fly ash is observed to have more uniformly spherical particles. This also explains the low surface area of Sugozu fly ash (Table 4).

The XRD patterns of the fly ashes are shown in Fig. 4. In general, the fly ashes consist predominantly of an amorphous phase together with crystalline mullite, quartz, and hematite. Compared with the Catalagzi and Iskenderun fly ashes, the Sugozu fly ash shows lower apparent intensities for several crystalline peaks, particularly those associated with mullite and hematite, suggesting lower apparent crystallinity under the present test conditions. Based on this qualitative observation, and when considered together with the SEM observations, particle size distribution, surface area measurements, dissolved Si and Al contents, and strength results, this may be associated with a higher amorphous phase content in the Sugozu fly ash.

Figure 5 illustrates the dissolved Si and Al quantities after immersing the fly ashes in a 10 mol NaOH solution at 75 °C for 4 h. As can be seen, the amounts of dissolved Si

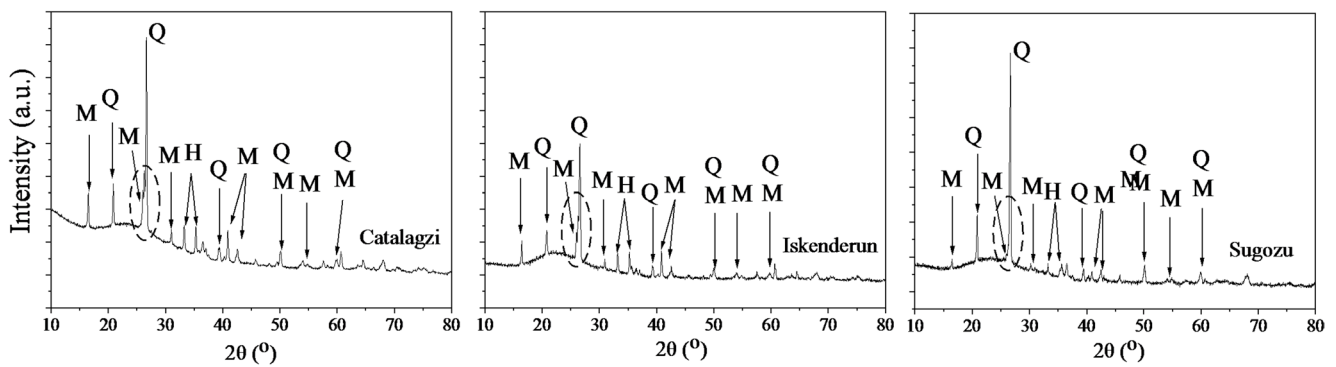


Fig. 4 XRD patterns of fly ashes; M=mullite, Q=quartz, H= hematite

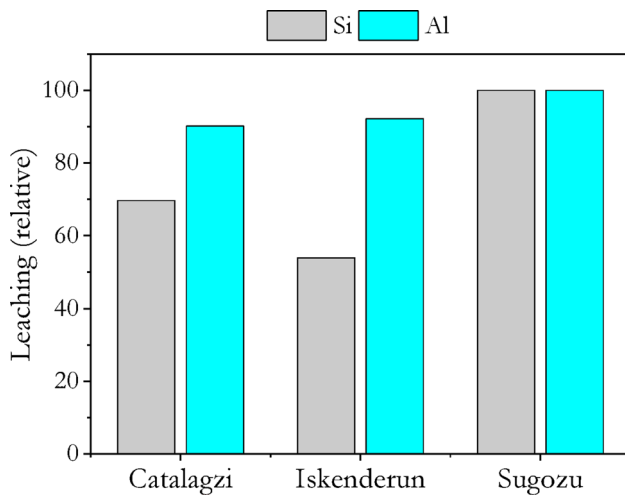


Fig. 5 Dissolved Si and Al amounts of three different fly ashes in NaOH solution

and Al for Sugozi fly ash are higher than those of the other two fly ashes. This indicates that Sugozi fly ash contains higher quantities of reactive Si and Al.

Based on the combined evaluation of LPSD and nitrogen adsorption analyses, SEM images, XRD patterns, and Si/Al solubility in NaOH solution, Sugozi fly ash is concluded to be more reactive than the other two fly ashes. Fly ashes with different reactivities are also expected to respond differently to varying curing temperatures and durations.

Compressive strength

Strength development of Catalagzi FGPs over 90 days

The 3, 7, 28, and 90-day compressive strengths of Catalagzi FGPs cured at ambient temperature were 0.0, 2.8, 21.2, and 28.2 MPa, respectively. These results indicate that the strength development of Catalagzi FGPs cured at ambient temperature progresses slowly [40, 53]. Slow strength gain is due to the slow alkali activation of fly ash at low temperatures. Studies have shown that the alkali activation of fly ash

in NaOH solution at 20 °C can be approximately 70 times slower than at 60 °C [54, 55]. Consequently, slow geopolymerisation at low temperatures causes very slow strength gain and low ultimate strength, as seen in Catalagzi FGPs. The low ultimate strength at ambient-curing highlights the importance of elevated temperatures for activating low-calcium fly ashes [38, 56, 57].

The 90-day compressive strength development of Catalagzi FGPs cured at different temperatures and durations is presented in Fig. 6. Note that the 3-day strength of 7-day cured specimens is assumed equal to that of 3-day cured specimens. As seen from Fig. 6, the compressive strength of the specimens that are cured for 8 h increases significantly over time. Specifically, the strength of the specimens cured for 8 h at the temperatures of 60 °C, 75 °C, and 90 °C rise 214.5%, 125.1%, and 119.8% from 3 days to 90 days, respectively. In contrast, specimens cured for 3 and 7 days show little increase, especially after 28 days.

The fresh FGP paste consists of fly ash particles with alkali activator-filled voids. The reaction products formed by the geopolymerisation fills these voids. As a result, the voids are partially filled with geopolymer gels. The degree of filled voids has a major impact on the strength and durability of FGPs. The progress in the formation of geopolymerisation products, which decrease the number of voids, is directly affected by temperature. At higher temperatures, reactions are faster, causing rapid solidification and high early strength. However, rapid solidification forms larger pores, resulting in a less ordered structure [53, 58, 59]. Although curing at 90 °C enhances early strength, the degree of geopolymerisation stabilises at levels similar to those achieved at lower curing temperatures over time. Consequently, the less ordered structure dominates at later stages, limiting the final compressive strength [58, 60, 61].

As shown in Fig. 6, Catalagzi FGPs cured at 90 °C for 3 days or longer achieved significant strength within the first 3 days, with only limited additional gain afterward. Consequently, their ultimate strengths were lower than those cured at 60–75 °C for the same period. Furthermore, the higher the

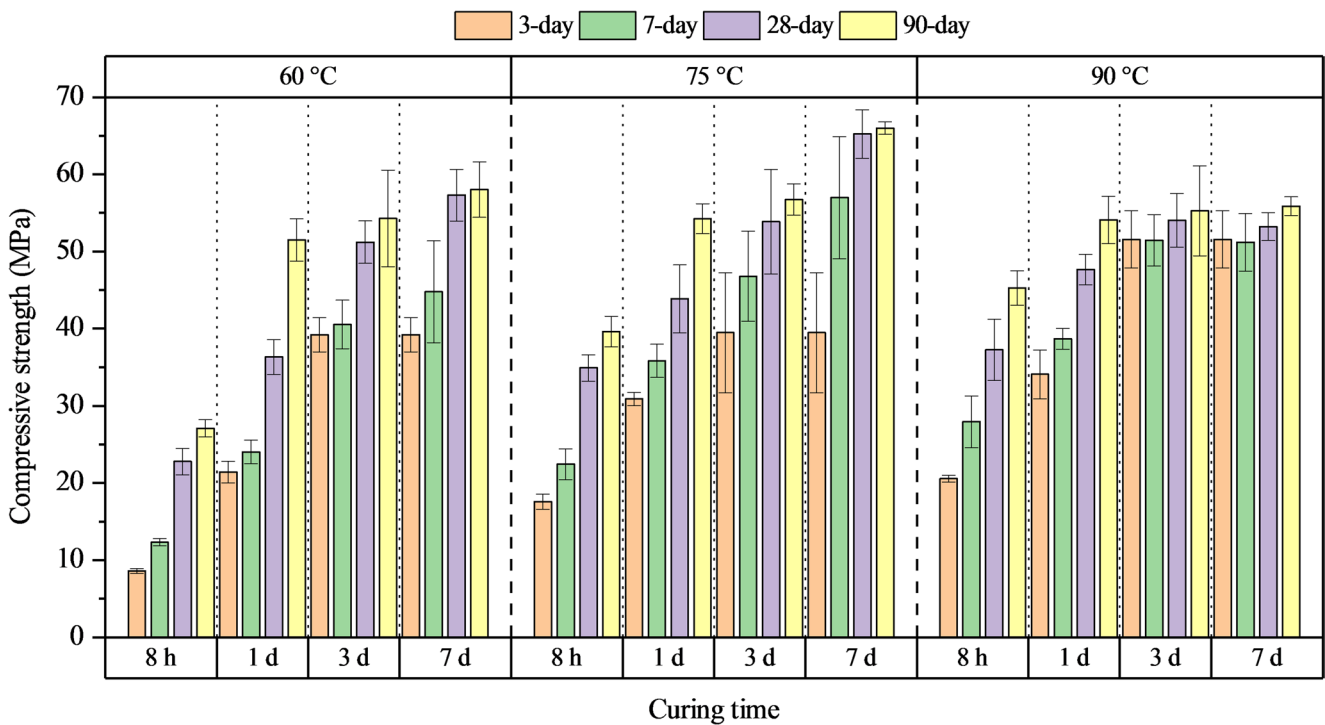


Fig. 6 Compressive strength development of Catalagzi FGPs under various curing temperatures and durations

reactivity of an aluminosilicate source, the more sensitive it becomes to the negative effects of elevated curing temperatures. It may also be adversely affected by relatively lower curing temperatures (as discussed in Sect. 3.2.2). Therefore, it should be noted that the curing temperature at which different fly ashes are negatively affected varies depending on their reactivity. For Catalagzi fly ash (and others with similar reactivity), a moderate curing temperature of 60 °C to 75 °C is required to achieve sufficient activation and to form a more uniform, less porous matrix with well-distributed reaction products. These FGPs also exhibit higher frost resistance due to reduced permeability [23].

In terms of curing duration, it is well-known that extending the thermal curing time increases the formation of reaction products and enhances the degree of cross-linking in the three-dimensional aluminosilicate network [62]. This leads to improvements in the strength. However, 28-day compressive strength results show that at 90 °C, extending curing beyond 24 h yields only marginal gains and may cause strength loss after 72 h. Similar observations have been reported in other studies [24, 40, 63]. This suggests that Catalagzi FGPs (and similar fly ashes) nearly complete their geopolymerisation within 28 days when cured at 90 °C for 24 h or longer. The strength loss observed beyond 72 h can be attributed to the accelerated release of water from the geopolymeric network at elevated curing temperatures due to the higher reaction rate [36], which may generate internal voids and promote shrinkage cracking caused by

drying shrinkage [24, 64, 65]. It should also be noted that with highly reactive fly ashes, the threshold curing duration at high temperatures—beyond which strength loss occurs—decreases (Sect. 3.2.2).

For Catalagzi FGP, samples cured at 90 °C for ≥ 3 days achieve very high strength quickly, indicating that 3 days at 90 °C is optimal for early strength. On the other hand, considering the early age of 7 days for concrete, curing at 75 °C for 7 days can be considered optimal, providing a strength of 57.0 MPa. However, for structures where impermeability is as critical as strength, curing at 60 °C for 7 days may be more suitable [36, 58]. These findings suggest that the optimal curing conditions for FGPs depends on specific application requirements, such as achieving early high strength, long-term durability, low porosity, or energy and cost savings [36].

28-day strengths of Catalagzi, Iskenderun, and Sugozi FGPs

The 28-day compressive strengths of Catalagzi, Iskenderun, and Sugozi FGPs cured at ambient temperature were measured as 21.2 MPa, 22.7 MPa, and 34.3 MPa, respectively. Additionally, Fig. 7 presents the 28-day compressive strengths of FGPs under varying curing temperatures and durations. At the mildest curing condition (60 °C for 8 h), the compressive strengths of Catalagzi and Iskenderun FGPs, at 22.8 MPa and 23.2 MPa respectively, exhibit

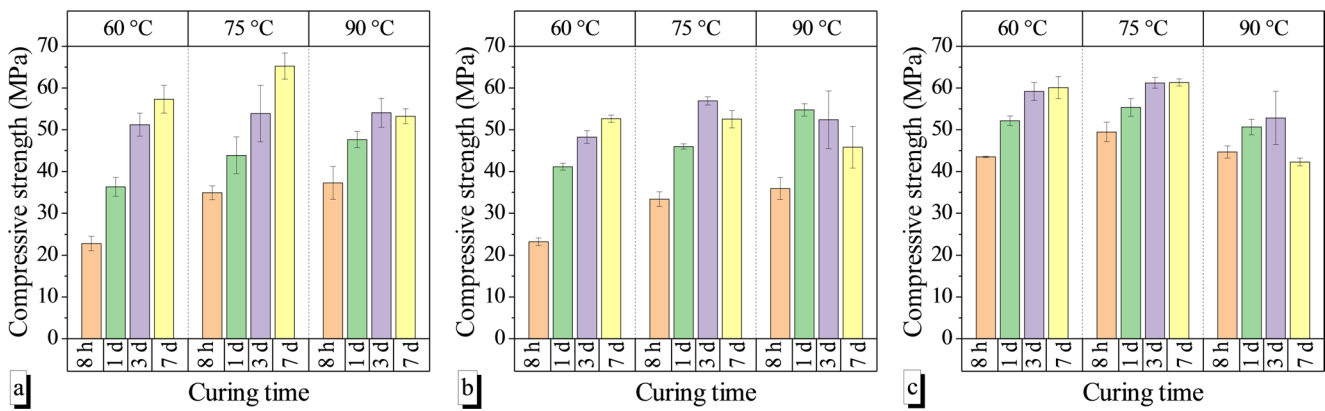


Fig. 7 Compressive strength at 28 days of Catalagzi (a), Iskenderun (b), and Sugozi (c) FGPs cured at different temperatures and durations

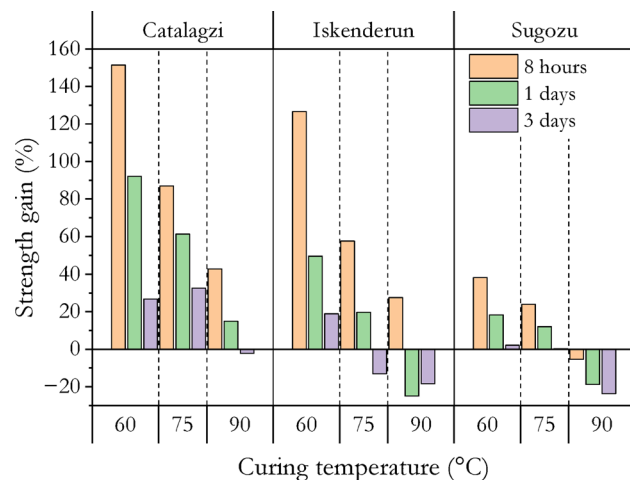


Fig. 8 Percentile compressive strength gain comparison of FGPs cured for 8 h, 1 day, and 3 days (compared to those cured for 7 days)

little difference from those cured at ambient temperature. In contrast, the Sugozi FGP shows a 26.8% increase, reaching 43.5 MPa. Sugozi FGP achieves higher strength at ambient temperature compared to the other two and exhibits significant strength improvement even under low thermal curing conditions. This suggests that the Sugozi fly ash, which is more reactive than the other two, requires relatively less thermal curing. Furthermore, the ability of Sugozi FGP to achieve high strength under other mild curing conditions (60 °C for 1 day, 75 °C for 8 h, 75 °C for 24 h, and 90 °C for 8 h) supports this conclusion.

Compressive strength of all three FGPs generally increases with longer curing at 60 °C and 75 °C, though the rate differs among fly ashes. To illustrate this, Fig. 8 compares the strengths of samples cured for 8 h, 1 day, and 3 days relative to those cured for 7 days. As observed, Catalagzi and Iskenderun FGPs cured for 7 days show much higher strength than those cured for 8 h. Specifically, for Catalagzi FGPs at 60 °C, 75 °C, and 90 °C, the increases are 151.4%, 86.9%, and 42.9%, and for Iskenderun FGPs,

126.7%, 57.5%, and 27.5%, respectively. It is evident that the influence of prolonged curing on strength improvement diminishes with higher temperatures. This outcome is supported by a study conducted by Sajan et al. [39], which stated that the geopolymerisation reactions of fly ashes are primarily influenced by curing temperature, and curing durations exceeding 3 days at temperatures above 60 °C do not result in a significant increase in strength. However, at 75 °C, this applies to Iskenderun FGPs but not Catalagzi FGPs (Fig. 8). Additionally, Atis et al. [11] noted that curing at 75 °C for over 2 days may reduce FGP strength. Similarly, Sugozi FGPs show limited strength gain with prolonged curing, even at low temperatures. Furthermore, extending curing from 8 h to 7 days at 90 °C increases strength in Catalagzi and Iskenderun FGPs, but reduces it in Sugozi FGPs.

This variability, also observed in other studies (e.g. [32]), is primarily attributed to the reactivity of the fly ash, aside from factors such as the concentration of the alkaline activator. Sugozi FGP, with higher reactivity, achieves high strength even at low temperatures and short curing times. In contrast, Iskenderun and especially Catalagzi FGPs require higher temperatures and/or longer curing. For instance, to reach 42.5 MPa, Catalagzi and Iskenderun FGPs need 60 °C for ≥ 3 days or 75–90 °C for ≥ 1 day, whereas Sugozi FGP reaches the same strength with only 8 h at 60 °C.

Although the chemical compositions of the three fly ashes are quite similar, the strengths of these FGPs indicate that their curing requirements for geopolymerisation vary. As discussed in Sect. 3.1, Sugozi fly ash contains a higher proportion of particles within the 1–100 μm size range, with a D_{90} value of 82.1 μm , indicating that about 90% of its particles are smaller than 100 μm . At the same time, Fig. 2 shows that the fraction of particles finer than 1 μm in Sugozi fly ash is relatively low (2.00%), suggesting that its particle size distribution is mainly concentrated within the 1–100 μm range. The abundance of particles in this size range may enhance the reactivity of aluminosilicate phases and therefore reduce the need for elevated curing temperatures [47].

Similarly, Gao et al. [66] reported that particle size distribution affects reaction activity more than amorphous content or average particle size.

One of the key advantages of fly ash particles being mostly smooth and spherical within a narrow size range is their relatively low total surface area. This characteristic reduces the amount of alkaline activator required to achieve adequate workability in geopolymers (Table 3). Since the alkaline activator is typically the most expensive component and one of the main contributors to the environmental impact of geopolymer systems, reducing the activator demand can significantly improve their economic and environmental performance [67–69]. Previous life-cycle and cost analyses have shown that alkaline activator production is the dominant driver of both the environmental impacts and production costs of geopolymer concrete [70–72]. Furthermore fly ashes rich in such particles also need lower curing temperatures and shorter durations, further reducing energy use and emissions [73]. Therefore, Sugozy FGPs are expected to be more cost-effective and environmentally friendly than those from other fly ashes.

Analysis of reaction products

Fourier-transform infrared (FTIR)

The chemical bonding of Catalagzi FGP cured at different temperatures and durations are characterised by FTIR. As can be seen in Fig. 9, the main FTIR bands of all FGPs are between 1300 and 800 cm^{-1} , indicating asymmetric Si-O-T (T = Si or Al) stretching [38, 74]. The main band at 1018 cm^{-1} in raw fly ash narrows and shifts to lower wavenumbers during alkali activation, showing dissolution of Si and Al monomers in early geopolymerisation. This process takes several days at ambient temperature or several hours at elevated temperatures. Since the FTIR spectrum in the study is

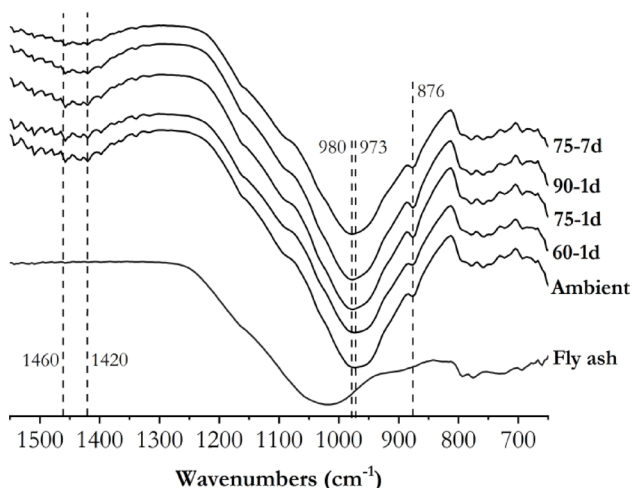


Fig. 9 FTIR spectra of raw Catalagzi fly ash and Catalagzi FGPs

obtained at 90 days, and considering that the later phases of geopolymerisation are nearly complete by this time, the following conclusion can be drawn [36]: The initial broad band of raw fly ash shifts lower due to the dissolution of Si and Al monomers from the raw material during the process from mixing to solidification. In the second phase, slight shifts to higher wavenumbers occur as silicate and aluminate oligomers polymerise. In the final phase of geopolymerisation, the formation of Si-rich cross-linked tectosilicate gel is anticipated to shift the main bands to lower wavelengths and eventually reach wavelengths below those of raw fly ash. Evaluating FGP spectra shows that increasing curing temperature and duration generally shifts the main band to higher wavenumbers: 973, 974, 977, 978, and 980 cm^{-1} for the AMB, 60-1d, 75-1d, 90-1d, and 75-7d, respectively, indicating more cross-linked geopolymer gels [29, 36, 39].

Weak bands between 1460 and 1420 cm^{-1} observed in the FGPs—but not in raw fly ash—indicate asymmetric O–C–O stretching vibrations [24, 75]. These bands show that excess Na^+ , which does not participate in geopolymerisation, reacts with atmospheric CO_2 to form Na_2CO_3 . This band is more prominent in samples cured at ambient temperature and at 90 °C. At ambient temperature, lower reaction progress leaves more unreacted Na^+ and insufficient filling of voids, and the higher permeability allows CO_2 to penetrate and increase carbonation. At 90 °C, high carbonation is linked to increased permeability caused by voids formed due to water evaporation (see Sect. 3.2). In contrast, the reduced band intensity in samples cured at 75 °C for 7 days likely reflects sufficient reaction product formation and lower permeability.

A band at 876 cm^{-1} , which is absent in the fly ash spectrum but present in FGPs, may indicate (i) Si–O terminal bond vibrations [38, 76], or (ii) the formation of sodium bicarbonate [29, 75].

Another band that is not present in the fly ash spectrum but appears in the FGP spectrum is located at 876 cm^{-1} . This may indicate two conditions: (i) Si–O terminal bond vibrations [38, 76], or (ii) the formation of sodium bicarbonate [29, 75]. The first suggests increased crystallinity of cenosphere and metal oxide particles, which do not participate in geopolymerisation [38, 46]. The second reflects carbonation resulting from exposure to atmospheric CO_2 [77].

X-ray diffraction (XRD)

Figure 10 shows the XRD patterns of raw Catalagzi fly ash and Catalagzi FGP cured at ambient temperature. The raw fly ash exhibits a predominantly amorphous phase together with crystalline quartz, mullite and hematite, whereas the ambient-cured FGP is characterised mainly by quartz- and mullite-related reflections. The main amorphous phase of

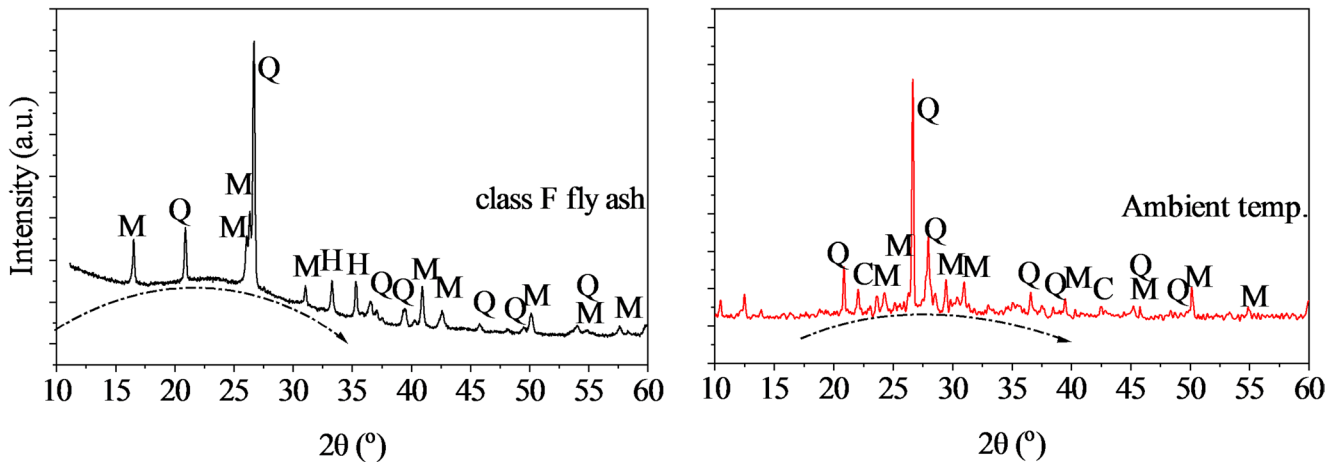


Fig. 10 XRD patterns of Catalagzi fly ash and Catalagzi FGP at ambient temperature; M=mullite, Q=quartz, H=hematite, C=calcite

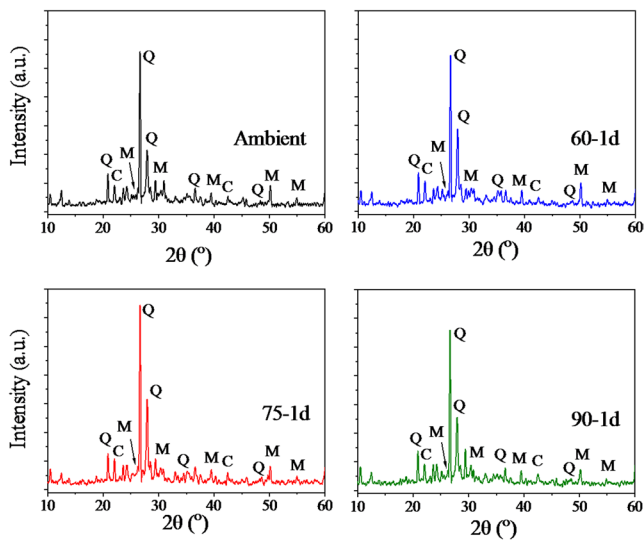


Fig. 11 XRD patterns of Catalagzi FGPs at ambient, 60 °C, 75 °C, and 90 °C; M=mullite, Q=quartz, C=calcite

the raw fly ash appears as a broad hump within the 2θ range of 15° to 35° (Fig. 10), which is modified in the FGP pattern. This may be attributed to the consumption of reactive constituents from the amorphous fly ash during geopolymerisation, in agreement with the observations reported by Bai et al. [65]. In addition, several mullite-related reflections in the FGP pattern show lower apparent intensities than those in the raw fly ash, while the hematite-related reflections around 33° and 35° are no longer clearly distinguishable. Given the resistance of mullite to alkali dissolution under the curing conditions considered here [38], these differences are interpreted as qualitative variations in apparent peak intensity within the diffraction pattern. Quartz-related reflections remain visible in the FGP pattern, which may also be influenced by the sand used in mortar production. Taken together, these results suggest that geopolymerisation alters

the diffraction pattern of the raw fly ash mainly through the consumption of reactive amorphous constituents.

XRD analysis was conducted to examine changes in the diffraction patterns with increasing curing temperature. Figure 11 shows the XRD patterns of Catalagzi FGPs cured for 1 day at ambient temperature, 60 °C, 75 °C and 90 °C. In comparison with Fig. 10, where the difference between the raw fly ash and the ambient-cured FGP is more evident, changes in the amorphous contribution are less clear when only the 1-day-cured FGP specimens are compared. This may indicate that, after 1 day of curing, the diffraction patterns remain broadly similar and that differences in reaction progress are still limited. Only minor changes are observed in the apparent intensities of some mullite-related reflections, while the more crystalline phases appear to remain comparatively stable under alkali conditions [38]. Quartz-related reflections remain visible despite the increase in temperature. This is consistent with Latifi et al. [78] and Turan et al. [4], who indicated that quartz behaves as an inert phase under alkaline conditions.

Figure 12 shows the XRD patterns of Catalagzi FGPs cured at 75 °C for 1 day and 7 days. With increasing curing time, only modest changes are observed in the relative intensities of several reflections. In addition, no clearly new crystalline phases are observed under the temperature conditions considered in this study. This is consistent with Duxson et al. [79], who reported that new crystalline phases are generally not expected at curing temperatures of 70–90 °C, whereas crystallisation may occur at higher temperatures, such as 120 °C.

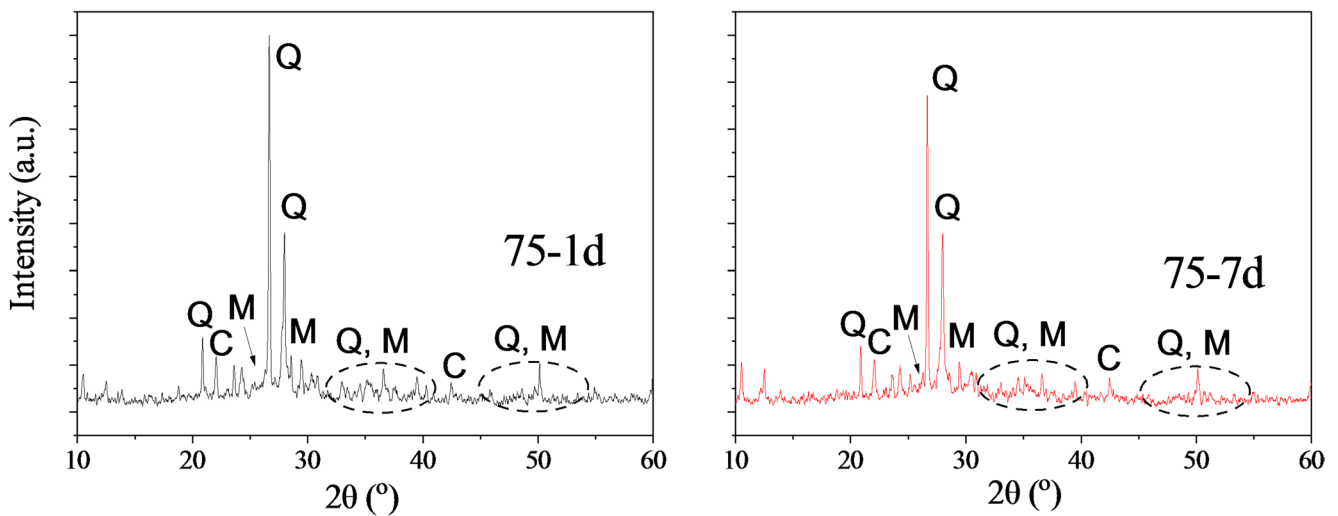


Fig. 12 XRD patterns of Catalagzi FGPs cured at 75 °C for 1 and 7 days; M=mullite, Q=quartz, C=calcite

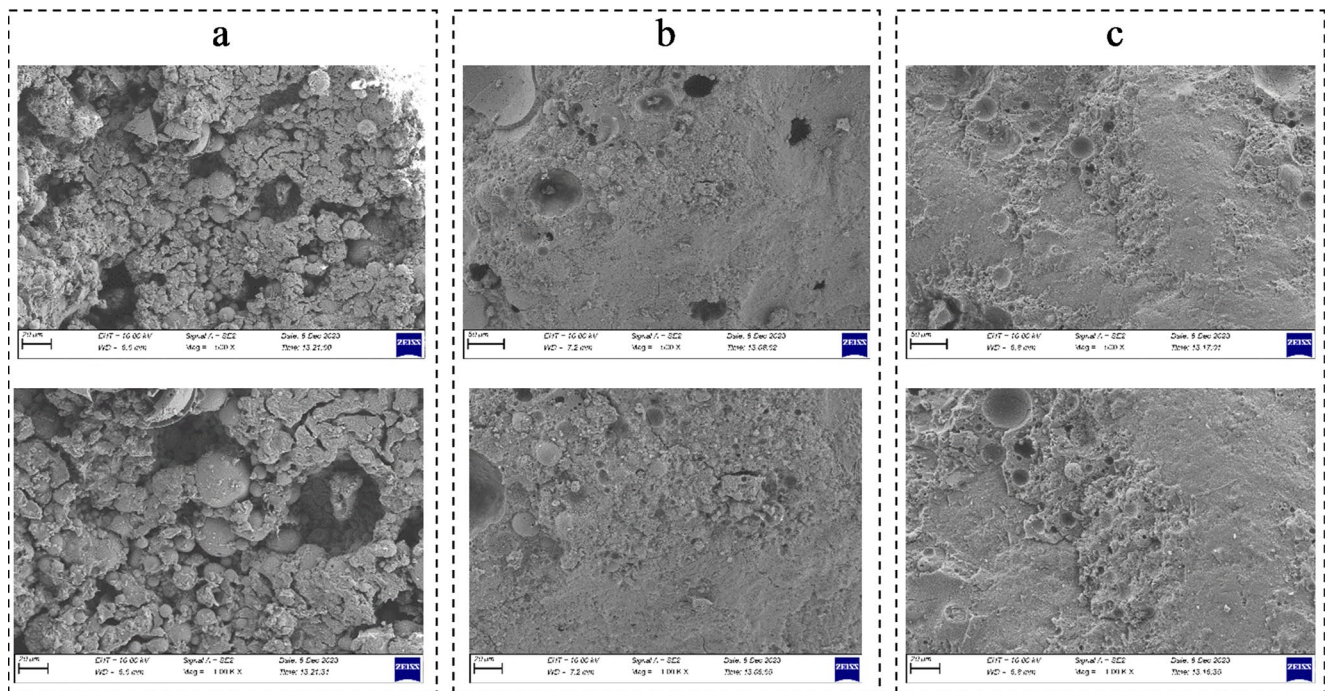


Fig. 13 SEM micrographs of Catalagzi FGPs at different curing conditions: (a) AMB, (b) 75-1d, and (c) 75-7d

Microstructural and Porosity Analysis

Scanning electron microscope (SEM)

The SEM micrographs of Catalagzi FGPs are shown in Fig. 13. Samples cured at ambient temperature contain many unreacted fly ash particles. These particles decrease at a curing temperature of 75 °C. This indicates that highly amorphous fly ash dissolves in a high-alkaline environment, and its consumption rises with temperature. On the other hand, enhanced curing increases the number of fly ash particles

with slightly reacted outer surfaces. In addition, Extending the curing duration to 7 days also increases these particles and the reaction rates on their surfaces. Thus, as curing temperature and duration increase, the outer glassy layers of the particles (thought to be cenospheres) join geopolymerisation reactions, while the crystalline outer layer stays intact [38, 46]. The low specific gravity of Catalagzi fly ash (2.13) indicates that many cenosphere particles, usually below 0.80 in specific gravity, exist in this raw material [46, 51, 80]. Although more fly ash particles react at higher curing temperatures and longer durations, some spherical particles

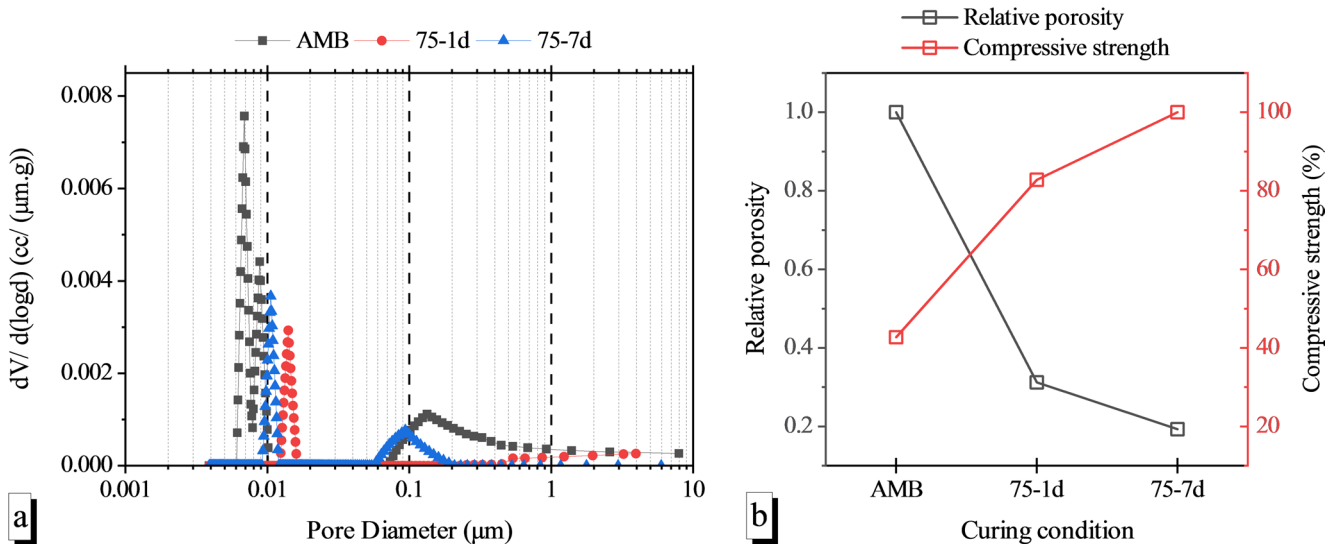


Fig. 14 MIP results of Catalagzi FGPs: (a) differential pore size distribution; (b) correlation between total porosity and compressive strength

remain unreacted. These inert metal-oxide particles, which do not participate in geopolymerisation, may act as micro-aggregates and help prevent microcracks [79, 81]. However, in some cases, these rigid particles may also cause cracks in the hardened paste [68].

It can be observed that higher curing temperature and time allow geopolymer gels to fill pores and voids, creating a denser matrix. Yilmaz et al. [23] reported similar results and stated that higher curing temperature and time accelerate reactions, affecting reaction products and microstructure. As a result, more homogeneous and compact matrices form. Oz et al. [82] also noted that a denser microstructure increases compressive strength. Thus, the compressive strength results of this study align with the SEM findings.

Mercury intrusion porosimetry (MIP) method

The polycondensation reactions in geopolymers are significantly influenced by curing time and temperature, as these factors alter the long-range arrangement of tetrahedral units. This change has a direct impact on the pore structure [83]. To observe the effect of the curing process on pore structure, MIP results of Catalagzi FGPs, which were also subjected to SEM analysis, are presented in Fig. 14(a). Furthermore, to establish a relationship between strength and porosity, the total porosity values obtained from this analysis are plotted against compressive strength in Fig. 14(b).

Pores in concretes are generally classified into four categories: micropores smaller than 10 nm, transitional pores between 10 and 100 nm, mesopores in the range of 100–1000 nm, and macropores larger than 1000 nm. Micropores are usually harmless, while transitional pores are considered slightly harmful. However, pores between 100 nm and 10

μm are linked to voids, cracks, and defects caused by unreacted particles, and are therefore considered detrimental [83–85].

As shown in Fig. 14(a), Catalagzi FGP cured at ambient temperature has a greater void volume for pores larger than 100 nm compared to heat-cured samples. This indicates that heat-curing reduces the size of larger voids. Zhang et al. [86] similarly reported that higher curing temperatures decrease the pore sizes in FGPs. Additionally, the total porosity of ambient-cured Catalagzi FGP is significantly higher than that of the heat-cured samples (Fig. 14(b)). This suggests that heat-curing promotes geopolymerisation, which reduces the size and connectivity of large voids.

On the other hand, extending the heat-curing duration at 75 °C from 1 day to 1 week slightly decreases the total porosity. This indicates that geopolymerisation reactions continue in Catalagzi FGP with longer heat-curing durations. As discussed in Sect. 3.2, the relatively low-reactivity of Catalagzi fly ash requires higher curing temperatures and longer durations for activation. In contrast, the high-reactivity of Sugozi fly ash achieves high strength at lower curing temperatures and shorter durations by completing (or maturing) its geopolymerisation. Considering the direct influence of pore structure on strength, it is expected that more refined pore structures can be achieved at lower curing temperatures and durations for highly reactive fly ashes like Sugozi.

Conclusions and future perspectives

In this study, Class F fly ash based geopolymer (FGP) mortars were produced using fly ashes obtained from three different thermal power plants. The mortars were cured at various temperatures and durations to evaluate their performances. The strength development of Catalagzi FGPs from 3 to 90 days revealed that samples subjected to short-term heat-curing showed slower strength gain, whereas those cured at 60 °C and 75 °C revealed a significant increase in compressive strength over time. In contrast, prolonged curing (3 and 7 days) at 90 °C did not result in substantial additional strength gain. FTIR and XRD analyses indicated that increasing the curing temperature and duration enhanced the dissolution of Si and Al from the fly ash and increased the degree of cross-linking within the geopolymeric gel. Moreover, SEM images and MIP results confirmed that this intensified geopolymerisation process contributed to the formation of a denser matrix with reduced porosity, ultimately resulting in improved compressive strength.

The reactivity of fly ash is a key factor influencing the heat-curing requirements of FGPs to ensure sufficient geopolymerisation. FGPs produced with Sugozy fly ash, which exhibits relatively higher reactivity than other two fly ashes, achieved high strength even at lower curing temperatures and shorter durations. In contrast, the other two FGPs required more demanding curing conditions to reach comparable strength levels. In addition, the surface area of fly ash, shaped by its particle size distribution and morphology, plays a decisive role in determining the alkaline activator demand of FGPs. Owing to the predominance of uniformly spherical particles within a narrow size range, Sugozy FGPs required less alkaline activator during production. The combined effect of reduced heat-curing and lower activator demand in Sugozy FGPs is expected to decrease both CO₂ emissions and production costs compared with the other fly ash sources.

Future studies should also incorporate quantitative phase analysis, such as Rietveld refinement, in order to provide a more rigorous evaluation of the crystalline phases and amorphous content of different fly ashes. This would strengthen the interpretation of XRD results and improve the assessment of fly ash reactivity. In addition, further research should explore the scalability and real-world applicability of FGP concretes through life cycle assessments (LCA) and cost-benefit analyses. Such studies are crucial for facilitating the transition of these materials from laboratory research to market-ready sustainable construction products.

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