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Degradation mechanisms of metakaolin-based geopolymers incorporating waste glass under combined low-temperature preparation and drying curing

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ABSTRACT

Due to the environmental drawbacks and performance limitations associated with cementitious materials, geopolymer materials have been viewed as the best alternative in the near future. The key advantages are low energy consumption and the incorporation of waste materials into its mixture design. However, the combined effect of low-temperature and drying conditions remain insufficiently understood. This study replicates these conditions in a laboratory setting by preparing geopolymer mortar at room temperature (10–15°C), subjecting it to two curing temperatures (23°C and 60°C) for 24 hours, and then exposing it to controlled oven curing at 23°C. Mechanical, transport, thermal, and microscopic tests have been conducted to gain a comprehensive insight into its performance. Soda-lime glass waste was selected as a partial substitute for metakaolin, given its amorphous nature and silica content, to assess whether its reactive properties could benefit the reaction under these conditions. This study reveals that low-temperature preparation retards the reaction, while excessive moisture loss hinders its development, limiting the contribution of glass under these conditions. As a result, the materials develop a heterogeneous microstructure rich in pores and microcracks. The results show that compressive strength and thermal conductivity decrease with increasing glass content, whereas water porosity, gas permeability, and chloride migration coefficient increase, indicating that the resulting microstructure facilitates easy transport pathways. Additionally, the microscopical observations showed that the formation of precipitated gel and crystalline products on the surface sealed cracks and pores, thereby increasing the apparent density; however, these secondary products didn't enhance the long-term performance of the geopolymers.

1. Introduction

Since the nineteenth century, cement has been the main binder in concrete, serving as a fundamental component in the growth of modern infrastructure [1]. However, its widespread, growing global use and rising demand for cement are linked to high energy consumption, heavy fuel use, depletion of natural resources, and substantial greenhouse gas emissions, all of which contribute to climate change and health problems such as respiratory diseases and lung cancer [2,3]. In the 21st century, where sustainable development has been viewed as a global priority, the continuous usage of both resource-intensive and

high-carbon-emissions products is increasingly recognized as unsustainable [4,5]. Accordingly, the United Nations has set sustainable development goals, urging the construction sector to reduce its environmental impact [6]. This has increased research into finding alternative materials to substitute for traditionally used cement-based material [7]. While partial substitution with supplementary cementitious materials or waste materials can be helpful, it does not fully eliminate reliance on cement usage [8–10]. As such, it is better to shift the focus toward complete replacement, especially the development of alkali-activated binders such as geopolymers [11,12]. These materials answer the sustainability problem because they are produced with low

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CO₂ emissions compared to OPC, use abundant waste materials, and diminish resource extraction [13].

Besides that, geopolymers display enhanced performance and better properties compared to traditional cementitious materials [14,15]. This is due to their reactive aluminosilicate network and the specific selection of ingredient proportions in their mix design [16]. The reaction initiates when an aluminosilicate rich precursor, such as metakaolin or fly ash, comes into contact with a sodium or potassium alkali activator [17,18]. This reaction forms a 3-dimensional amorphous gel network known as sodium aluminosilicate hydrate (N-A-S-H) or potassium aluminosilicate hydrate (K-A-S-H), which affects the overall mechanical properties of the resulting material [19]. Although geopolymers show good performance in most published studies, their practical use remains limited due to their sensitivity to curing conditions. It is well known that the geopolymerization reaction is strongly dependent on temperature, especially at higher curing temperatures, which promote the dissolution of aluminosilicate species and accelerate the formation of the amorphous gel [20]. Most research studies focus their curing temperatures ranging from ambient conditions (20–25°C) to 60–80°C to accelerate the reaction and achieve proper material development [21]. Conversely, these controlled laboratory curing conditions do not reflect real applications, where geopolymer or cement-based concrete are typically cast on-site and exposed to environmental conditions rather than regulated thermal conditions. In addition, moisture inside the reaction is crucial for the geopolymerization process [22]. Water is an important parameter as it is responsible for ion transport and gel formation, but its loss during drying can delay the chemical reaction and can negatively affect the resulting microstructure [23]. So, temperature and moisture shouldn't be considered independent parameters, as they both influence geopolymerization kinetics and the resulting microstructural development simultaneously. Although the impact of curing temperature, moisture, and glass powder has been individually studied in the literature, their combined effect on the geopolymerization reaction under low temperatures and drying curing is not well investigated, especially when it is prepared at temperatures as low as 10–15°C. In fact, it is important in research to not only focus on conditions that enhance geopolymer properties but also to understand their behavior in less ideal environments. This investigation provides valuable insights into the mechanisms behind strength development, porosity changes, and durability of geopolymer mortar.

In this research, waste glass powder is used as a partial substitution for metakaolin to investigate whether this addition persists under both low-temperature and drying conditions, since its effectiveness under these conditions remains unclear. This research introduces a non-classical curing methodology to evaluate the effects of low-temperature and drying conditions on a laboratory scale. Geopolymer mortars were prepared at a controlled room temperature of 10–15°C, subsequently subjected to oven exposure for 24 hours to facilitate two initial curing regimes at 23°C and 60°C, after which a drying phase was implemented to simulate realistic moisture loss scenarios. Soda-lime glass powder was used to partially replace metakaolin in the mix design at 0%, 10%, 20%, and 30% by mass. The study addresses the effect of preparation temperature and curing conditions on the gel evolution, mechanical performance, and overall reaction kinetics. Particular attention is paid to the evolution of durability-related properties, such as water porosity, gas permeability, chloride-ion penetration resistance, and thermal conductivity under the same preparation and curing conditions. Additionally, numerical microscopy was conducted to examine the microstructure of the resultant mortar by assessing the presence of pores and cracks and correlating these characteristics with the other properties evaluated in this study.

2. Materials & Methods

2.1. Material used

The following materials are the selected ingredients for this research:

- **Metakaolin** is the aluminosilicate rich raw material, which interacts with the alkali activator. The Metakaolin Argical M1000 was provided by IMERYS in France and was used as received, without any additional treatment, with a specific gravity of 2.6 g/cm³, a specific density of 0.4 and a BET surface area of 17 m²/g. Table 1 presents the chemical composition of metakaolin and Fig. 1 shows the crystalline phases present in metakaolin.
- **Glass powder** is chosen for this research to partially replace metakaolin due to its high silica content (73.28% by wt), as indicated in Table 1, and 100% amorphous content as illustrated by the XRD examination in Fig. 1. The type of glass, typically referred to as soda lime glass, primarily from beer and wine bottles, is collected from bars and clubs in La Rochelle, France. The bottles were placed in water for a few days, thoroughly cleaned, and to eliminate any remaining liquids, impurities, and adhesive paper. Following this, the glass was ground using a Retsch BB200® jaw crusher. Then, sieving was conducted to collect powder with particle sizes below 80 micrometers, ensuring the material's fineness for this study. Also, its specific gravity was measured to be 2.5 g/cm³.
- **Alkali activator** used is industrial-grade sodium silicate purchased from Xatico in France, having a 10-molar ratio. The used solution has a silicate modulus Na₂O/SiO₂ of approximately 1. Detailed information on trace impurities was not available from the supplier.
- **Sand** used in this study is a standardized sand supplied from Société nouvelle du Littoral (SNL), France.

2.2. Sample preparation

As this study investigates geopolymer preparation under low-temperature conditions, the materials used and the laboratory temperatures were maintained between 10 and 15°C before and during mixing. All geopolymer mortars were prepared using the same alkali activator and sand, with a liquid-to-solid (L/S) ratio of 1.28 and Si/Al = 1.5. The variable in the mix design was the substitution of metakaolin with glass powder, ranging from 0% to 30%, as summarized in Table 2. For geopolymer preparation, all solid raw materials (metakaolin, glass powder, and sand) were added to the mixer, followed by the alkali activator to produce the geopolymer mortar. The fresh mortars were then cast into their respective molds and vibrated to remove air bubbles. Within the first 24 h after casting, the mortars were cured at two different temperatures (23 °C and 60 °C). After this initial curing period, all samples were maintained under 100% ventilated drying oven conditions at 23 °C until the designated testing age.

2.3. Characterization method

The compressive strength test was conducted in compliance with EN 196-1 [24], using cubic 40 × 40 × 40 mm specimens. To ensure repeatability, five samples were tested on each of the 5 testing days (days 7, 24, 28, 90, and 180). At day 180, before the compressive strength test, 3 cubes for each batch were selected to measure their mass and dimensions to calculate their apparent density using Equation 1:

$$\rho = \frac{m}{v} \quad (1)$$

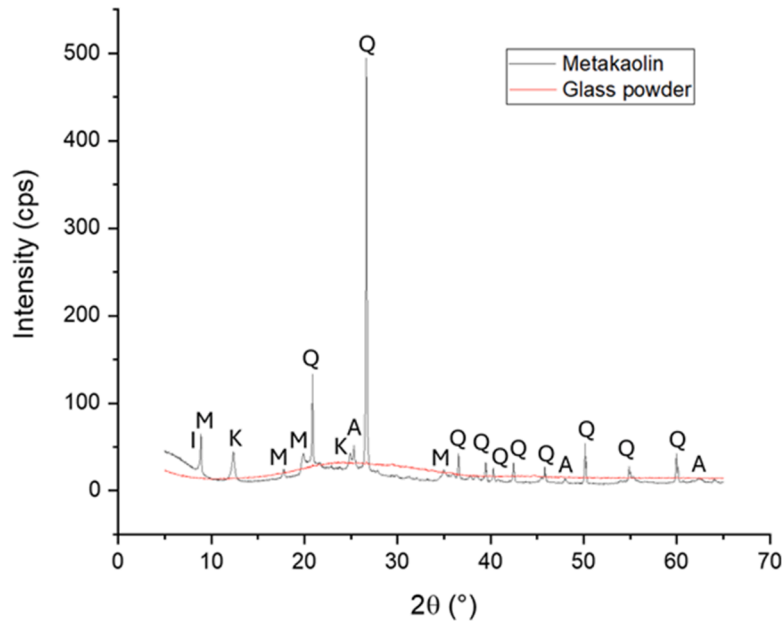
where ρ represents the apparent density in (Kg/m³), m the mass of the specimen in (Kg) and v its volume in (m³).

The water porosity test was conducted in accordance with NF P 18-459 [25] on 40 × 40 × 20 mm samples, and, for repeatability, three samples were selected for each batch on the 4 testing days (days 7, 28,

Table 1

The chemical composition of Metakaolin and soda lime glass powder.

Oxides (%)	SiO ₂	Al ₂ O ₃	K ₂ O	Na ₂ O	Fe ₂ O ₃	TiO ₂	CaO	MgO
Metakaolin	53.7	41.61	1.05	0.11	2.17	1.12	0	0.23
Glass powder	73.28	3.13	0.93	13.29	0	0	8.91	0.46

**Fig. 1.** XRD patterns of glass powder and Metakaolin, where the diffraction peaks are denoted with the corresponding index: I = Illite, K = Kaolinite, M = Muscovite, Q = Quartz, A = Anatase.**Table 2**

Proportions of the mixture design for 1 kg of total solids.

% of glass replacement	Metakaolin (g)	Waste glass (g)	Sand (g)	Alkali activator (g)
0%	250	0	750	320
10%	225	25	750	320
20%	200	50	750	320
30%	175	75	750	320

90, and 180). The porosity is calculated using the following equation 2:

$$\varnothing = \frac{M_{sat} - M_{dry}}{M_{sat} - M_{hyd}} \times 100 \quad (2)$$

where $\varnothing$ represents the water porosity in (%), M_{sat} is the saturated mass (in air) of the specimen in (g), M_{dry} is the 105°C dry mass of the specimen in (g), M_{hyd} is the hydrostatic mass (in water) of the saturated specimen in (g).

The gas permeability test was conducted according to the XP P18-463 [26] guideline using a Cembureau apparatus, with an 11 × 5 cm cylindrical sample. The samples were saturated in a desiccator for 24 hours prior to testing, and testing was performed at 180 days of curing. One sample was tested for each batch; yet, 6 measurements were taken at different volumes and pressures, and the reported value is the average of those measurements. Equation (3) is used to calculate the permeability coefficient using modified Darcy's law:

$$k_x = 2\mu L \frac{P_0}{P_0^2 - P_1^2} \frac{Q}{A} \quad (3)$$

where k_x represents the gas (air) permeability coefficient in (m²), μ is the viscosity of the gas (dry air) at 20 ± 2°C in (Pa × s), L is the thickness of the specimen in (m), P_0 is the inlet pressure and P_1 is the outlet pressure

measured in (Pa), Q is the flow rate in (m³/s) and A is the cross-sectional area of the specimen in (m²).

After performing the gas permeability test, the same samples were used to conduct the standard chloride migration test following the NT Build 492 standard [27]. The samples were under vacuum saturated with the same solution used at the downstream of the migration cell (anodic electrolyte). The cathodic electrolyte contains a 2 mol/L sodium chloride (NaCl) solution, and the anodic electrolyte contains a 0.3 mol/L sodium hydroxide (NaOH) solution. A constant polarization potential of 30 V was maintained for all samples during 24 hours. Then, the specimens were split in half, and the cutting surface was sprayed with a 0.1 mol/L silver nitrate solution to determine the chloride penetration depth, as shown in Fig. 2, for two samples representing samples cured at 23 and 60°C. The non-steady chloride migration coefficient was subsequently calculated using Equation 4:

$$D_{nsm} = \frac{0.0239 (273 + T) L}{(U - 2) t} \left(x_d - 0.0238 \sqrt{\frac{(273 + T) L \cdot x_d}{U - 2}} \right) \quad (4)$$

where D_{nsm} is the chloride migration coefficient in (x 10⁻¹² m²/s), U is the applied potential in (V), T is the average of the initial and final temperature in (°C), L is the thickness of the sample in (m), x_d is the average penetration depth in (m), and t is the test duration in (s).

Given that no standardized procedures for geopolymers materials are currently established, most durability assessments are adapted from those developed for conventional cementitious materials. Consequently, specific tests, such as carbonation resistance, chloride penetration, sulfate attack, and Alkali-Aggregate Reaction (AAR), cannot be directly applied without limitations due to fundamental differences in chemistry and microstructure between these materials [28]. The chloride migration test is one such example because it relies on assumptions about pore-solution composition and reaction mechanisms that differ

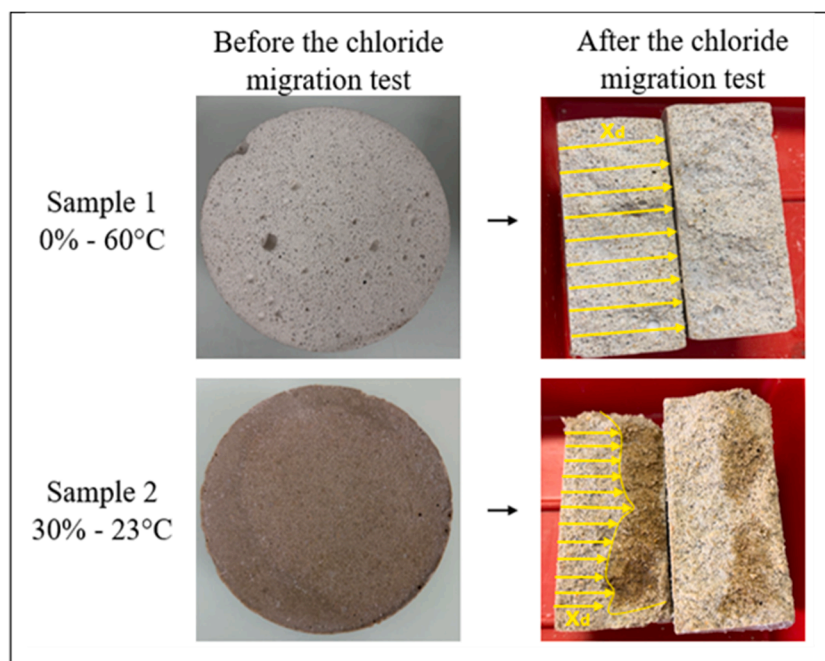


Fig. 2. Chloride penetration tests on different samples cured at 23 & 60°C, showing the before-and-after splitting and the addition of silver nitrate to measure the chloride penetration depth. It should be noted that sample 1 exhibits complete chloride penetration, whereas sample 2 exhibits partial penetration, as marked on the sample.

significantly in geopolymer systems [29]. In particular, the determination of chloride penetration depth using the silver nitrate colorimetric method may be affected by the high alkalinity of the pore solution, leading to the formation of both silver chloride (AgCl) and silver oxide (Ag_2O) [30]. In this case, silver ions can react with both chloride ions and/or hydroxyl ions. Consequently, the observed color change does not necessarily correspond strictly to the actual chloride penetration [31]. This can lead to inaccurate results for geopolymers when conducting this test, compared with traditional cementitious materials. Yet, this method has been widely employed in the literature for alkali-activated materials, primarily for comparative evaluation of mixtures tested under identical conditions rather than for absolute quantification of chloride transport properties [32].

Also, at 180 days of curing age, thermal conductivity was measured according to NF EN ISO 22007-2 [33]. This test was conducted using a Hot Disk Thermal Constants Analyzer TPS 1500 machine on 9.5 cm-diameter, 1.6 cm-high disc samples. For each test, two specimens at the same replacement level were created, as each test requires two samples of the same dimensions, with a sensor between them. The samples were taken directly from the oven to ensure conditions consistent with the other investigated tests.

Additionally, on the same samples, numerical microscopy was performed using a Keyence VHX-7000 microscope to assess morphology at all curing ages (7, 14, 28, 90, and 180 days). Based on the results, it was necessary to perform EDS analysis of the observed pink-pigmented particles and crystals to better understand the sample performance. The apparatus used was an Environmental SEM-FEI Quanta 200 ESEM/FEG coupled with EDAX GENESIS Energy Dispersive Spectroscopy (EDS) at an accelerating voltage of 20 kV and a water vapor pressure of 1.5 mbar. In addition, the chemical composition of metakaolin and glass powder was calculated from the elemental composition obtained by EDS to determine the molar composition of the oxides listed in Table 1.

The XRD presented in figure 1 for the raw materials were performed using a Bruker D8 Advance equipped with the Standard Drift Detector (SDD) LynxEye XE-T, calibrated on the $\text{K}\alpha_1$ radiation of copper. The powdered samples were scanned over the $5^\circ - 80^\circ 2\theta$ range with a step size of 0.05° .

3. Results

3.1. Water porosity

Fig. 3 shows the evolution of water porosity in geopolymer specimens with varying levels of glass substitution, measured at curing ages from 7 to 180 days. Initial curing was conducted for 24 hours at either 23°C or 60°C, after which the specimens were stored in a ventilated oven at ambient temperature. Specimens with 30% glass substitution in mass showed a greater tendency to fracture, which explains the large standard deviation, but these results were included to enable a full comprehensive comparative analysis.

Generally, the water porosity test results showcased a consistent increase with an increase in curing age and the percentage of glass substitution combined. This shows that the curing method is the major factor affecting porosity, as similar findings were observed across all mixture compositions, irrespective of the mass % glass substitution and initial curing temperatures examined. In particular, the curing phase led to significant water evaporation, which typically elevates porosity and induces shrinkage due to dehydration, thereby resulting in a more heterogeneous matrix rather than a homogeneous one [34]. Additionally, the relatively low initial temperature contributed to an increased porosity, as it retards the reaction and leaves unreacted particles within the geopolymeric system. It is also evident that a similar porosity trend was seen for the samples cured initially at 23°C and 60°C, which shows that even at 60°C, a 24 h curing temperature known for accelerating the geopolymerization reaction and promoting gel formation; the reaction remained incomplete, though it increased with age [35].

It is also important to note that adding glass powder as an additional silica source within the matrix did not enhance the reaction under these conditions; rather, porosity increased with increasing glass level and with longer curing periods at the two specified temperatures of 23 and 60°C. According to the literature, adding waste glass powder to geopolymers decreases the system's porosity [36]. Still, it has also been demonstrated that in waste glass-based geopolymer concrete, the content of unreacted silica and leaching within the geopolymer system tends to increase as the glass powder content rises, which will in turn lead to

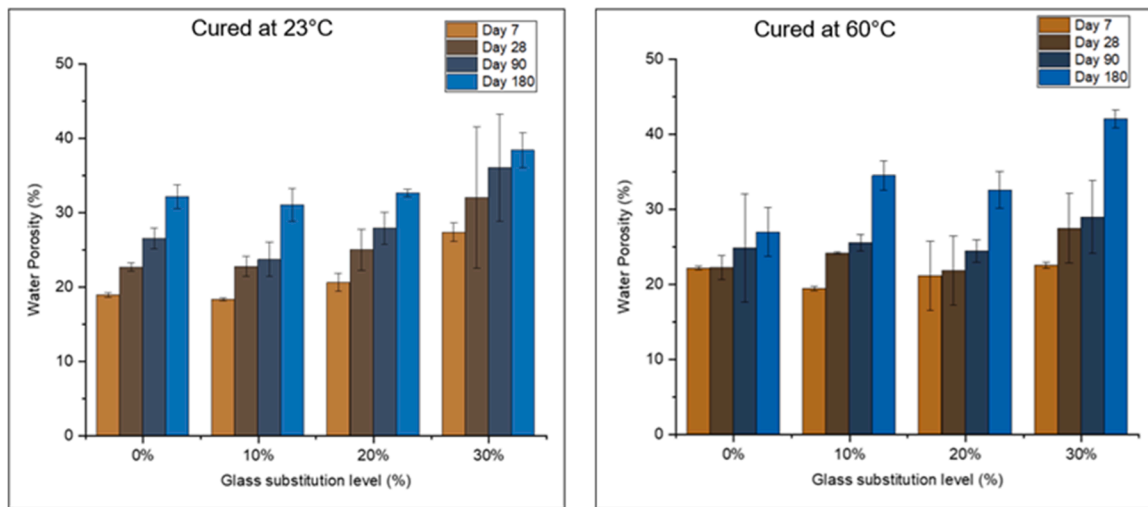


Fig. 3. Water porosity of geopolymer mortars where glass powder partially replaces metakaolin under different curing temperatures and days.

higher porosity across all ages, indicating incomplete reactions, which adversely affect the material's structural integrity and durability [37].

Finally, the combination of slow reaction kinetics and excessive moisture evaporation promoted the development of an open pore network, resulting in increased porosity and a less compact geopolymer matrix.

3.2. Numerical microscopy

Figs. 4–8 display numerical microscopy observations of all geopolymer samples (0–30% glass mass replacement) at two distinct curing ages. Observations were conducted on each testing day; however, for samples with 0% to 20% glass mass replacement, observations in this study began on Day 28, as minimal changes were noted on Days 7 and 14. Conversely, for samples containing 30% glass mass replacement, significant changes were observed on Day 7, so these images were subsequently included in this research article. Notably, for each curing day, the first row shows a 1000 μm scale to illustrate the general microstructure across all samples. The second row for each curing day presents a magnified view emphasizing features to facilitate understanding of microstructural changes; accordingly, the scale varies from 50 μm to 500 μm . Conversely, for samples with 0 to 20% replacement, since no substantial alteration was observed at Day 90 compared to Day 28, no additional magnified observations were incorporated. Conversely, for the 30% samples, it was considered important to include supplementary observations at day 90. In general, the figures demonstrate a very weak microstructure rich in numerous cracks and pores; hence, the microscope figures align with the water porosity test results, thereby clarifying that the heterogeneous matrix results from excessive drying and moisture loss.

For 0% samples, similar trends were observed across different curing temperatures. Pores are present at all curing ages; however, within these pores, crystals formed, as illustrated in figures 4b, 4g, and 4j. Nonetheless, figures 4d and 4i demonstrated that this method resulted in shrinkage upon reaching day 180 of curing. It was noteworthy that, for samples cured at 23°C, internal shrinkage within the pores was observed; conversely, for samples cured at 60°C, crystals appeared within the pores. For the 10% samples, a minor variation between the two curing methods was observed. The microscope observations show that samples cured at 23°C show evident visible pores that appears interconnected with cracks (figures 5a, 5c, and 5d). At day 28 (figure 5b), no crystals were detected within the pores; they were present surrounding the pores. By day 180 (figure 5e), crystals emerged within the pores. In contrast, for the samples cured at 60°C, no visible cracks were

present at any curing age in figures 5f, 5h, and 5i. At day 28 (figure 5g), crystals were observed within the pores. Subsequently, by day 180 (figure 5j), cracks measuring 100 μm were visible, with crystals present within the crack structures.

Similarly, the 20% specimens, those cured at 23 °C, showed pores and interconnected pores in figures 6a, 6c, and 6d. At 28 days (figure 6b), crystals were observed within the pores, but when reaching 180 days (figure 6e), crystals were present within the pores and on the surface as well. However, no cracks were visibly detected for the samples cured at 60 °C at any curing stage in figures 6f, 6h, and 6i. On the other hand, at day 28 of curing (figure 6g), crystals were observed within the pores along with a gel-like substance. Afterward, by day 180 (figure 6j), the pores were nearly completely filled. An interesting observation was made in the 30% samples at both curing temperatures (Figs. 7 and 8), where pink-pigmented particles resembling gel and crystals are visible throughout the whole microstructures. Initially, the microstructure clearly exhibits microcracks and pores at all curing ages. Figures 7b, 7d, 7f, and 7g illustrate the evolution of gel formation and crystallization: first within the pores, then within the cracks, and ultimately with crystals occupying the pores entirely and covering the surface at higher curing ages. Similarly, Fig. 8, which represents the 30% samples cured at 60°C, exhibits a comparable trend of evolution to that depicted in Fig. 7. Nonetheless, the magnified images particularly elucidate how the combination of crystals with the gel effectively seals the cracks and pores, as evidenced in Figures 8b, 8d, 8f, and 8g.

To better understand the nature of such products, an EDS analysis was conducted on samples after 180 days to determine the chemical composition of both the crystalline features and the pink particles. The results shown in Figures 7k and 8k confirm that the pink zones are predominantly silica-rich, whereas the crystalline products are calcium-rich, as the Figures 7l and 8l show a spectrum containing the matrix elements silicon (Si), aluminum (Al), oxygen (O), and sodium (Na) along with minor calcium (Ca) content. So, the pink-pigmented particles correspond to a silicon-rich phase, likely arising from the redistribution and reprecipitation of dissolved species from the glass powder, since metakaolin has no calcium content, as illustrated in Table 1. The overall microstructural reorganization is explained in Fig. 9, showing the coexistence of crystalline products (Fig. 9a) and the silicon-rich gel phase (Fig. 9b), concluding in the final gel product (Fig. 9c). The gradual evolution of this silicon-rich phase, which becomes more prominent over time, mirrors ongoing microstructural reorganization within the geopolymeric system. Also, their presence was seen more within pores and cracks, indicating that these regions serve as conducive sites for precipitation, and after that, they continue to cover the surfaces.

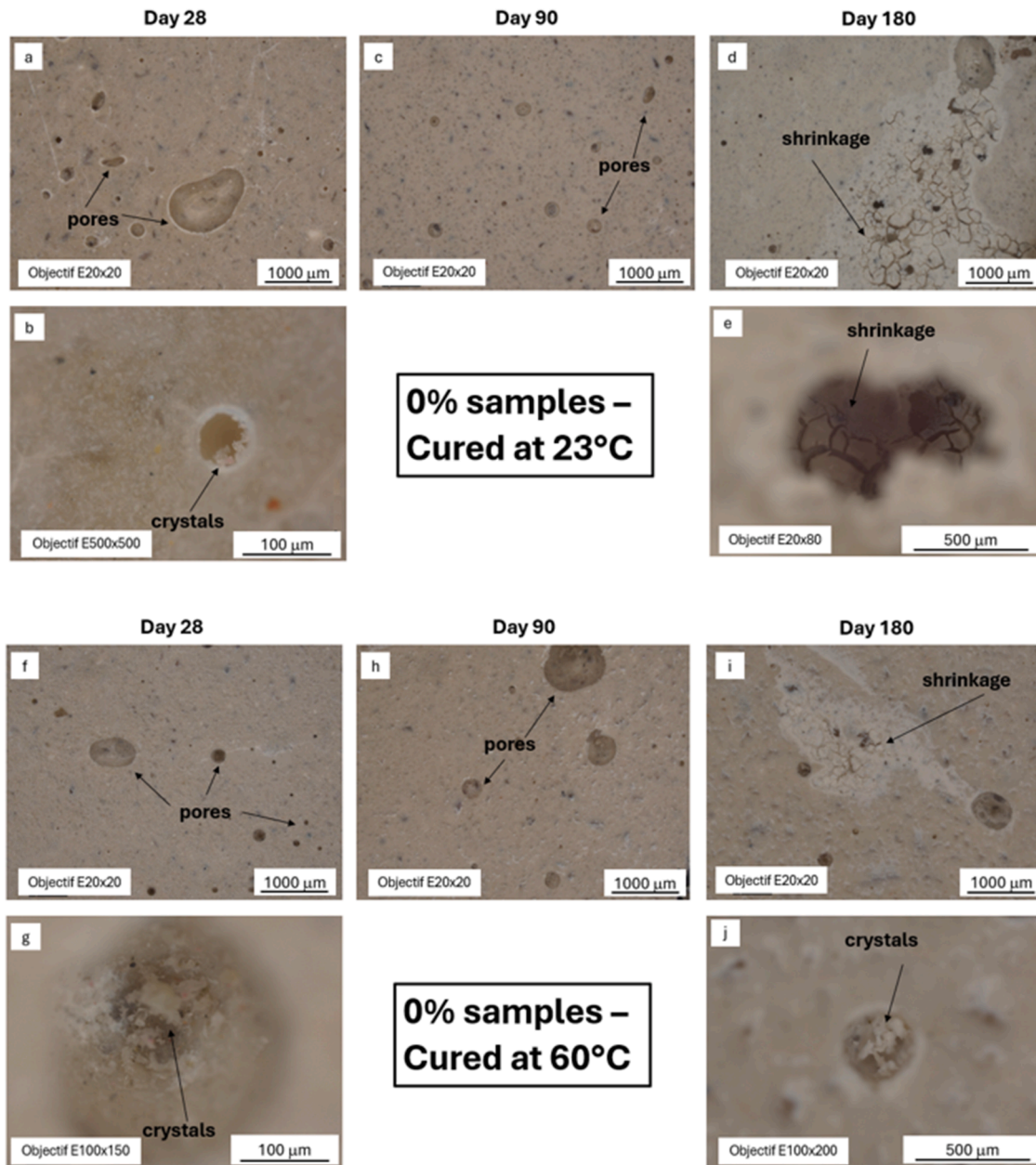


Fig. 4. Numerical microscopy images of geopolymer mortar with 0% glass replacement with metakaolin cured at 23°C (a-e) and 60°C (f-j) at different curing days: (a,b,f,g) 28 days, (c,h) 90 days, and (d,e,i,j) 180 days. Images (b) and (g) correspond to magnified views of the regions indicated in (a) and (f), respectively, at 28 days. Images (e) and (j) correspond to magnified views of the regions indicated in (d) and (i), respectively, at 180 days.

Simultaneously, the presence of calcium-rich crystalline products designates partial calcium segregation within the system. As depicted in Table 1, the glass powder contains 8.91% calcium oxide, whereas metakaolin contains none. Subsequently, increasing the glass content adds calcium availability in the matrix and modifies the Ca/Si ratio. It is documented in literature that there is a complexity of calcium-containing reaction products, and that also aligns with the present observations, in which the coexistence of silicon-rich and calcium-rich domains is clearly visible at the microstructural level [28]. In this context, the localized increase in the Ca/Si ratio may facilitate the formation of more ordered calcium-containing regions within the matrix, consistent with other studies that found that amorphous geopolymer gels can evolve toward more crystalline calcium-silicate-hydrate (C-S-H) structures when sufficient calcium is present [38]. However, this interesting behavior appears to remain localized and does not uniformly modify the matrix, as observed in the tests conducted in this research.

Moreover, it was observed that glass addition influences the microstructural evolution: the samples containing 0% glass exhibit primarily pores, with significant shrinkage only after 180 days, whereas samples containing 10-30% glass demonstrate earlier microcrack formation, indicating an easy reaction pathway. This suggests that the additional Si and Ca content introduced by the glass powder led to internal stress and microstructural instability during prolonged drying. In spite of this, their presence becomes more pronounced in samples where glass is replaced by 30% metakaolin for both curing temperatures, indicating secondary products formed at later curing ages. These illustrations are also consistent with previous research studies done on metakaolin-based geopolymers, indicating that it may undergo partial crystallization and gradual degradation over time, in contrast to Ca-rich systems such as fly ash-based materials, which tend to develop more stable microstructures with time [39,40]. This means that although calcium is introduced into the reaction via glass powder, its quantity and distribution remain

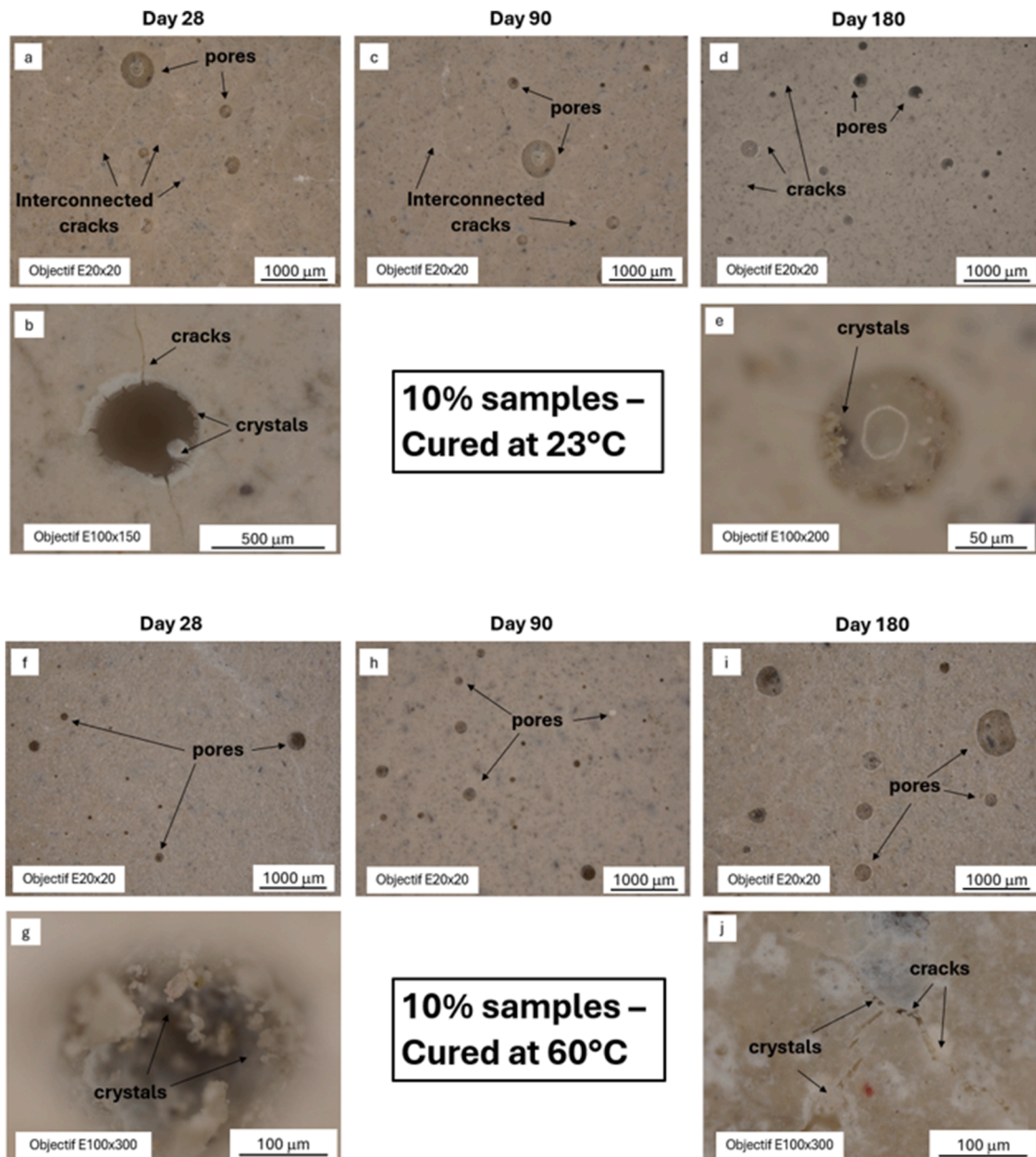


Fig. 5. Numerical microscopy images of geopolymer mortar with 10% glass replacement with metakaolin cured at 23°C (a-e) and 60°C (f-j) at different curing days: (a,b,f,g) 28 days, (c,h) 90 days, and (d,e,i,j) 180 days. Images (b) and (g) correspond to magnified views of the regions indicated in (a) and (f), respectively, at 28 days. Images (e) and (j) correspond to magnified views of the regions indicated in (d) and (i), respectively, at 180 days.

insufficient to stabilize or modify the reaction under the curing conditions developed in this study. Table 3 summarizes the microstructural evolution of the various geopolymer samples, without focusing on the initial curing temperatures, as no noticeable differences were observed. It should be noted that early ages are assumed for days 7 and 14 of aging, whereas later ages are assumed for days 28, 90, and 180.

3.3. Compressive strength

Fig. 10 depicts the variation in compressive strength relative to glass powder substitution and curing temperature at five distinct aging periods (days 7, 14, 28, 90, and 180). It shows a consistent decrease across all samples, indicative of ongoing structural degradation under these conditions. It is noteworthy to mention that the influence of curing at 60°C during the initial curing days doesn't show an early strength gain, indicating that the thermal treatment for 24 hours is inadequate for

accelerating the slow chemical reactions induced by low preparation temperature. Additionally, the results indicate that compressive strength decreases with increasing glass content across all curing ages which in turn lead to an increase in the porosity. This hypothesis is supported by existing literature, which states that these two properties are proportional; as compressive strength increases, porosity decreases [41]. Furthermore, these results are consistent with microstructural observations, revealing a heterogeneous microstructure with interconnected cracks and pores, which accounts for the decrease in compressive strength. The presence of such defects adversely impacts the mechanical performance of geopolymers by serving as stress concentration points that promote crack propagation, thereby ultimately diminishing their strength.

Although gel restructuring and reorganization were seen, particularly by day 180, they did not contribute to a mechanical enhancement of the structure. To gain further insight, an apparent density test was

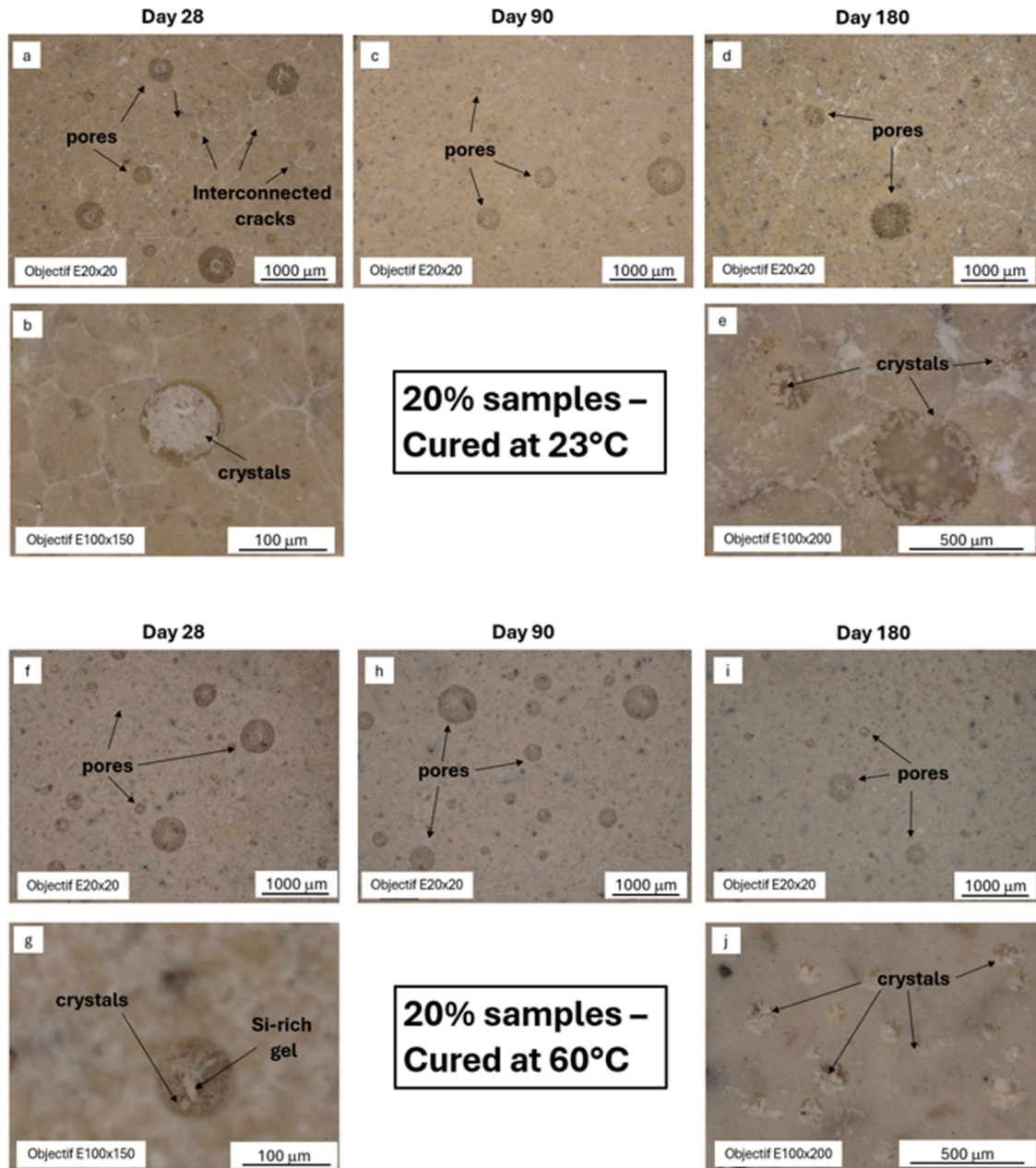


Fig. 6. Numerical microscopy images of geopolymer mortar with 20% glass replacement with metakaolin cured at 23°C (a-e) and 60°C (f-j) at different curing days: (a,b,f,g) 28 days, (c,h) 90 days, and (d,e,i,j) 180 days. Images (b) and (g) correspond to magnified views of the regions indicated in (a) and (f), respectively, at 28 days. Images (e) and (j) correspond to magnified views of the regions indicated in (d) and (i), respectively, at 180 days.

performed at day 180, with the results presented in Fig. 11 compared with the compressive strength results at the same day of curing.

In general, a decrease in density is associated with reduced compressive strength, as lower density typically indicates increased porosity [42]. Conversely, this study showed that as the glass content increases, the apparent density increases while the compressive strength decreases under both curing conditions (23°C and 60°C). The increase in density may be associated with the precipitation of gel and crystalline products, which seal cracks and pores, but the findings showed that it didn't affect the continued decline in compressive strength on day 180 of curing. Although this observation is supported by numerical microscopy images, further investigations are needed to understand this behavior, such as mercury intrusion porosimetry (MIP) testing, which allows distinguishing between gel-filled pores and microcracks, and testing the compressive strength beyond day 180. Since not all samples

show 100% filling at day 180, the reaction is ongoing and still undergoing restructuring and reorganization. On the other hand, the samples without glass content showed the highest compressive strength despite their moderate density, suggesting greater structural integrity and homogeneity than the 10-30% glass-containing samples.

3.4. Gas permeability

Fig. 12 shows the gas permeability coefficient at 180 days of curing for all geopolymer specimens at different glass replacement levels and curing temperatures (23°C and 60°C). It can be observed that the permeability coefficient increases with higher glass content across both curing conditions. For samples cured at 23°C, the gas permeability coefficient increased by one order of magnitude with 10%-20% glass replacement, followed by a moderate increase at 30% glass replacement.

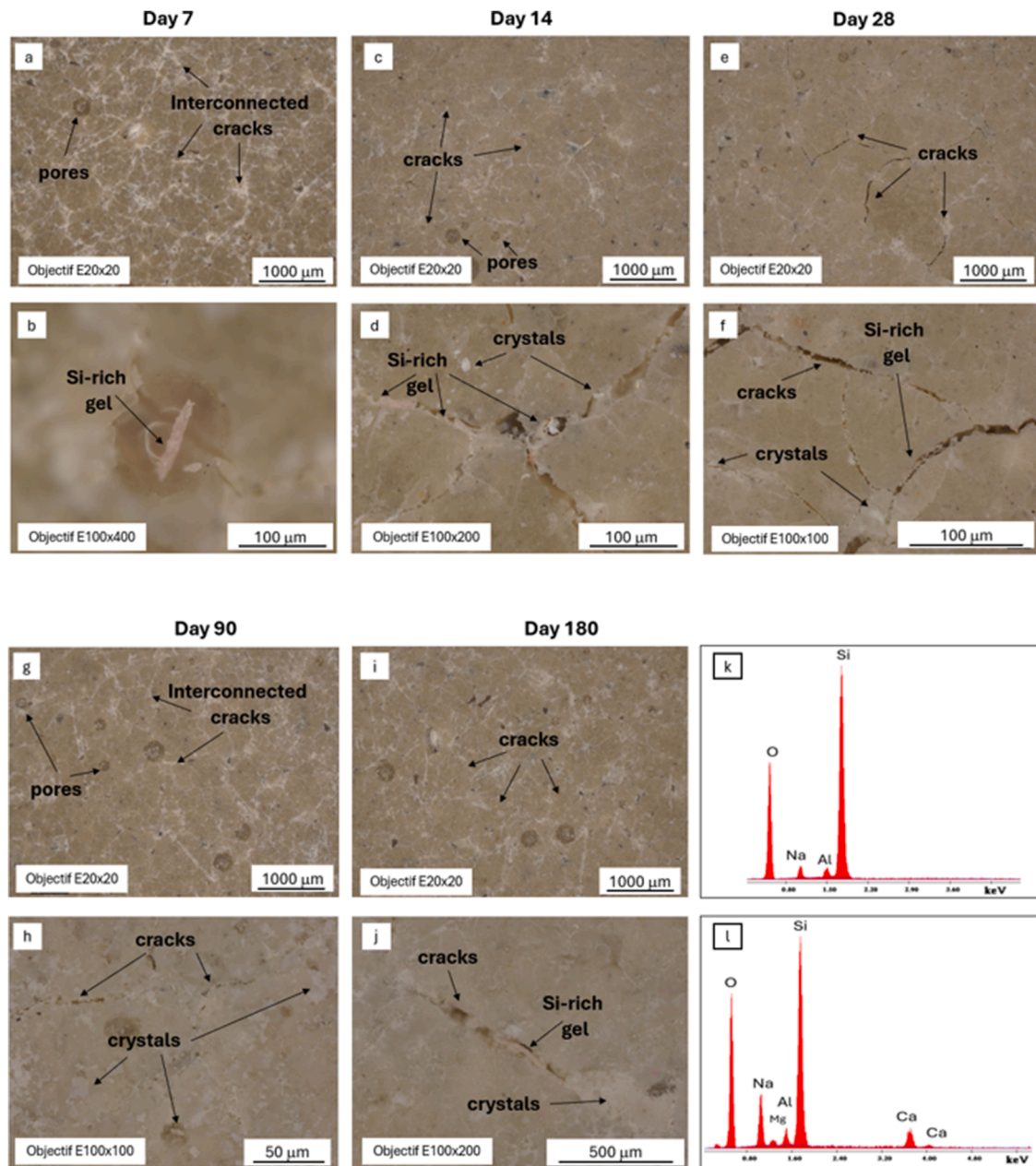


Fig. 7. Numerical microscopy images of geopolymer mortar with 30% glass replacement with metakaolin cured at 23°C at different curing days: (a,b) 7 days, (c,d) 14 days, (e,f) 28 days, (g,h) 90 days, and (i,j) 180 days. Images (b,d,f,h,j) correspond to magnified views of the regions indicated in (a,c,e,g,i), respectively. Images (k) and (l) correspond, respectively, to the EDS spectra at 180 days of the pink particles and crystals detected.

By contrast, for samples cured at 60°C, a slight decrease was observed between 10% and 20%, and an increase of one order of magnitude was observed at 30% glass replacement. Also, minimal changes were anticipated as the glass content replacing metakaolin increased for the same curing method. This test aligns well with porosity and microstructural analyses, indicating that gas transport is primarily governed by pore-network connectivity rather than by the mere presence of pores in the microstructure. Despite the fact that porosity plays a crucial role in gas flow, permeability primarily depends on the presence of channels that allow air to pass through pores and cracks. Isolated pores can contribute minimally to permeability, but even a slight increase in crack connectivity significantly improves gas flow.

Also, an interesting difference in curing temperatures was detected. Samples cured at 23°C had permeability values on the order of 10^{-14} m^2 , whereas those cured at 60°C had 2 orders of magnitude lower (10^{-16} m^2). This variation can be explained by microstructural observations, which

showed interconnected cracks in the 23°C cured samples, establishing continuous pathways that facilitate gas flow. On the contrary, although pores are present in the 60°C cured samples, the absence of extensive crack networks demonstrates a less-connected pore structure, resulting in decreased permeability value compared to the samples cured at 23°C. Additionally, the increased complexity of transport pathways caused by the discontinuous pores in the 60°C samples, except for those with 30% glass replacement, hinders gas movement and decreases permeability, even though pores are present.

This approach is supported by literature, which states that permeability is influenced by pore connectivity rather than total porosity, and that interconnected pores significantly enhance transport properties [41]. Furthermore, although gel reorganization at 180 days resulted in pore and crack filling, this process did not substantially reduce gas permeability. It therefore implies that the reaction products have decreased the pore volume but do not impact the continuous channels

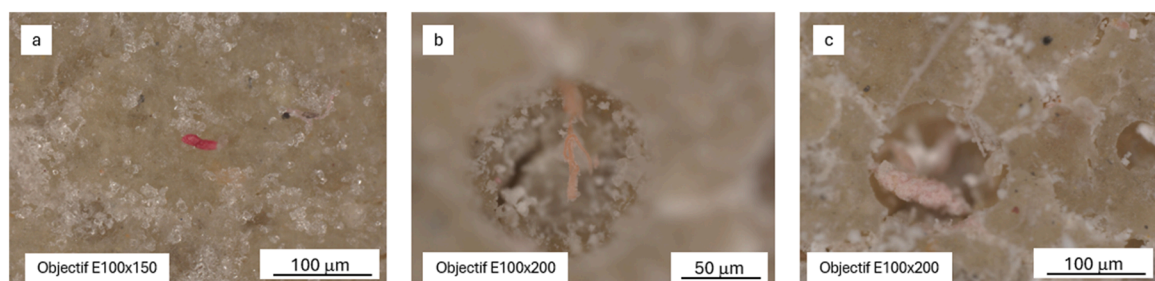
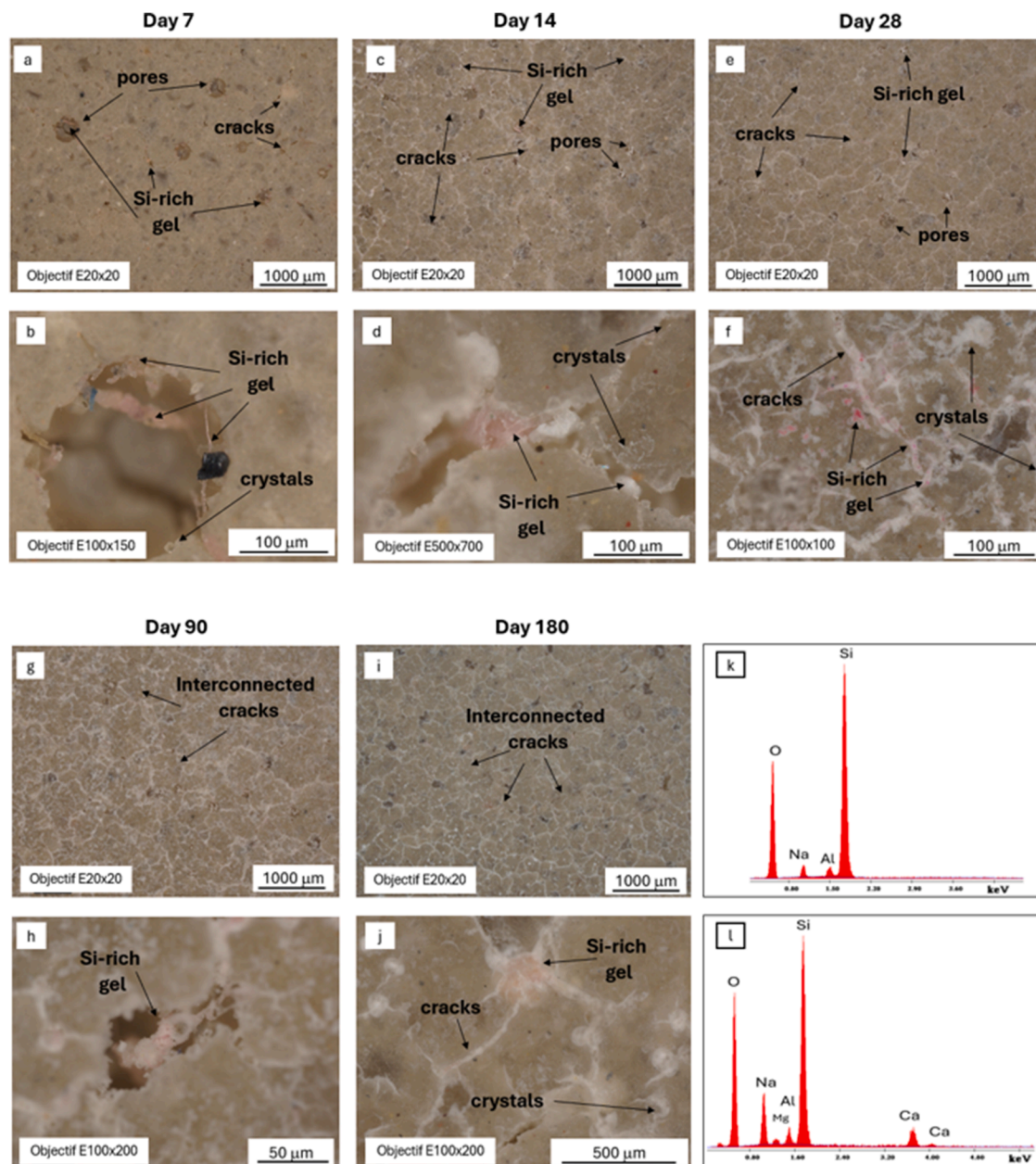


Fig. 9. Numerical microscopy images illustrating the evolution of the microstructure of geopolymer samples with 30% glass replaced, demonstrating: (a) the matrix containing calcium-rich crystalline products; (b) a pore region containing crystalline products and pink-pigmented silicon-rich gel products; (c) the coexistence of calcium-rich crystalline and silicon-rich gel products within the matrix.

Table 3

A summary of the microstructural evolution of all geopolymer samples at different glass substitution levels.

% of glass replacement	Cracks	Pores	Ca-rich crystals	Si-rich gel	Microstructural evolution
0%	Visible as shrinkage at 180 days	Visible in all ages	Formed within pores only	Not present at all ages	Occurrence of shrinkage and formation of crystals
10%	Visible at later ages	Visible in all ages	Formed within pores and cracks	Not present at all ages	Growth of crystals inside pores and cracks
20%	Visible at later ages	Visible in all ages	Formed within pores and cracks	Present at later ages	Pore filling becomes more noticeable
30%	Visible at all ages	Visible in all ages	Formed within pores, cracks and surface	Present at all ages	Gradual sealing of pores and cracks

necessary for advective gas transport. This advocates that such gel reorganization remains local and does not stop the continuity of transport pathways within the microstructure. Unlike typical trends reported in the literature [41] where continued reactions reduce porosity and permeability over time, the conditions employed in this research restricted such improvements, and transport remains primarily affected by drying-induced microstructure resulting from prolonged oven curing.

3.5. Chloride ion migration

Fig. 13 shows the non-steady chloride migration apparent coefficient for all geopolymer mortars cured at 23°C and 60°C. Generally, coefficients for both temperatures exhibit similar trends, indicating consistent chloride transport behavior across all samples at both curing temperatures.

The lowest values are seen for the 10% glass substitution for both curing temperatures, indicating the best resistance to chloride penetration at this level. Nevertheless, beyond 10%, both coefficients increase, particularly at 20% and 30%, signifying reduced resistance to chloride penetration at a higher glass replacement rate. This is consistent with the porosity and gas permeability results, which showed that a higher glass content produces a more interconnected pore structure and increased

transport flow, thereby confirming that chloride transport is significantly affected by the accessible pore structure. This indicates that incorporating porosity into the diffusion coefficient does not alter the overall transport behavior; it simply highlights the effect of pore structure on transport mechanisms. This emphasizes that both pore connectivity and total porosity influence ion migration, with connectivity remaining the predominant parameter, as previously confirmed by the gas permeability results. While gas permeability relies on advective transport, chloride transport relies on ionic transport through the pores. However, despite this difference, both types of transport depend on pore network accessibility. Microscopy illustrations further support this statement, revealing that interconnected cracks, especially at higher glass substitution levels, offer easy pathways for ion migration. In addition, although gel reorganization at later curing ages partially refilled pores and cracks, this process was not sufficient to hinder chloride transport, similar to the gas permeability results.

Also, it has been realized that curing conditions have a distinct influence on transport properties. Although the samples cured at 60°C show lower values at 10% glass addition, they showed a significant increase at higher glass substitution levels, surpassing those of the samples cured at 23°C. This phenomenon points to the fact that, although initial thermal curing may facilitate early reactions, it does not prevent the development of transport pathways at later curing days, particularly under excessive drying conditions.

3.6. Thermal Conductivity

Fig. 14 represents the thermal conductivity results for all geopolymer specimens cured at 23°C and 60°C, when they reached 180 days. It can be concluded that as the glass content increased, thermal conductivity decreased for both curing conditions, with marginally lower values for samples cured at 60°C. The decrease in thermal conductivity results from increased porosity as the glass content increases. An increase in the number of pores means more air within the structure, and given that air exhibits lower thermal conductivity compared to the solid phase of the geopolymer, this results in a decrease in thermal conductivity. Consequently, improving the efficiency of heat transfer through the samples. The results obtained range from approximately 1.4 to 1.9 W/m·K, which is within the typical range for conventional structural concrete.

The thermal conductivity results correlate well with previously observed tests, such as increases in porosity and transport properties, as well as heterogeneous microstructure observations, improving the thermal properties of the materials. In conclusion, although the inclusion of glass powder provides a modest improvement in thermal performance, geopolymer mortars incorporating glass powder are mainly

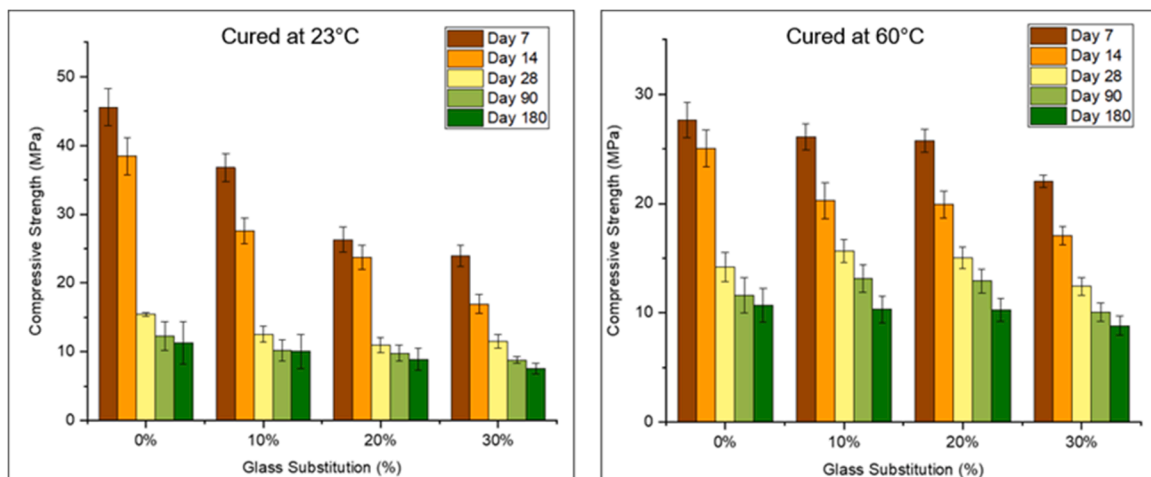


Fig. 10. Compressive strength results of geopolymer mortars at two curing temperatures (23°C and 60°C).

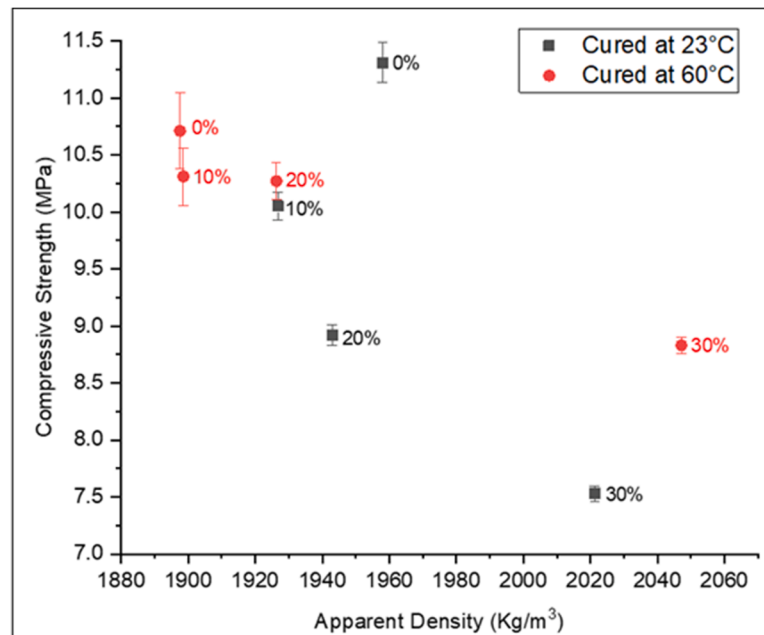


Fig. 11. Compressive strength vs apparent density of geopolymer mortars at two curing temperatures (23°C and 60°C) at day 180 of curing.

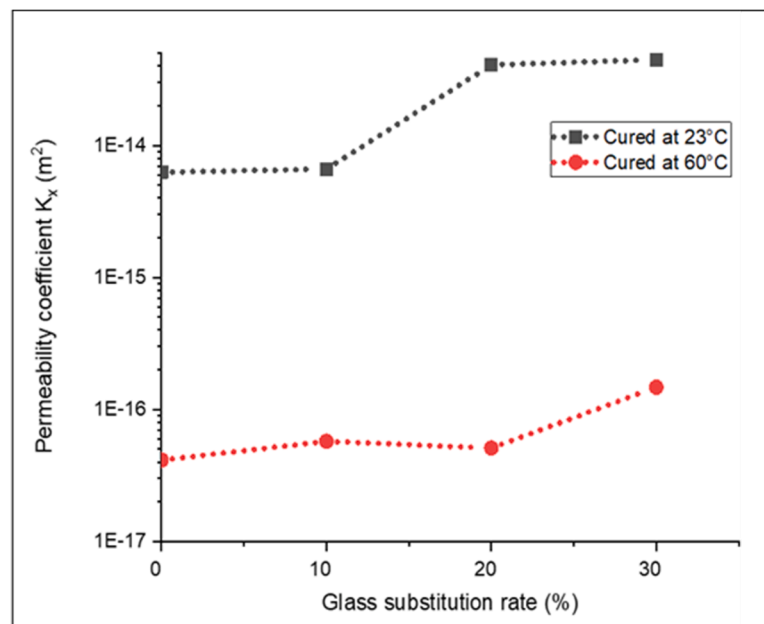


Fig. 12. Gas permeability coefficient of geopolymer mortars at two curing temperatures (23°C and 60°C).

suitable for traditional structural applications rather than for insulation.

4. General discussion

The experimental investigations conducted in this study have shown that the curing method affected the microstructural mechanism, which in turn controlled the mechanical and durability performance of the geopolymer. In other words, the reaction kinetics have affected the reaction mechanism, although not directly tested in this study. But the experimental tests, such as compressive test, porosity, numerical microscopy, gas permeability, chloride ion diffusion, as well as thermal conductivity, proved this mechanism as follows:

4.1. Effect of curing conditions on reaction kinetics

Geopolymerization is a reaction between metakaolin and 100% amorphous glass powder when mixed with sand and an alkali activator. The reaction usually begins with an equilibrium among its reactive species: alumina, silica, and calcium, all dissolved by sodium. This dissolution is followed by polycondensation and amorphous gel formation, which together produce a hardened geopolymer. This is the normal reaction mechanism when metakaolin is the source material, as it is typically prepared at ambient temperature. However, these reaction kinetics are strongly affected by temperature and moisture content. In this study, temperature was lowered during the preparation period, and then the samples were transferred to an oven at two different curing temperatures (ambient and elevated) for 24 hours to assess normal and

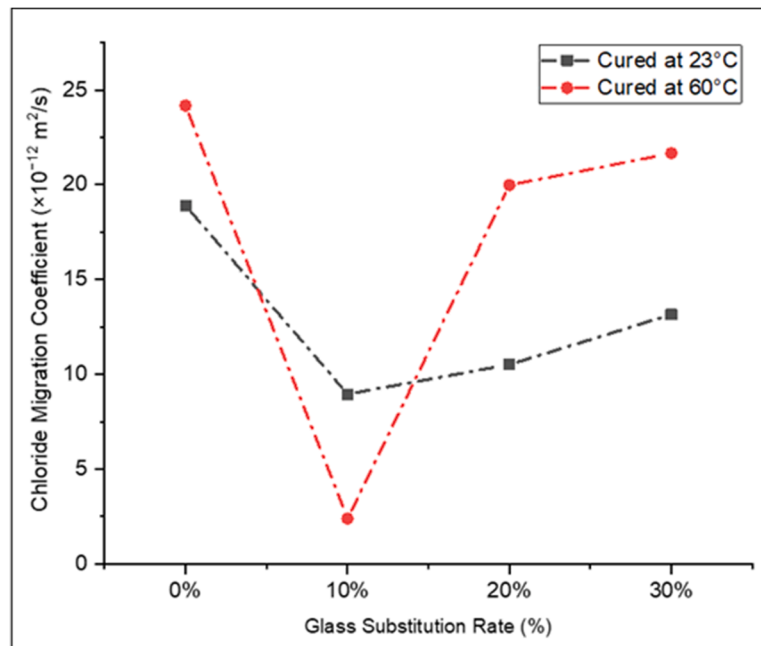


Fig. 13. Chloride migration coefficient of geopolymer mortars at two curing temperatures (23°C and 60°C).

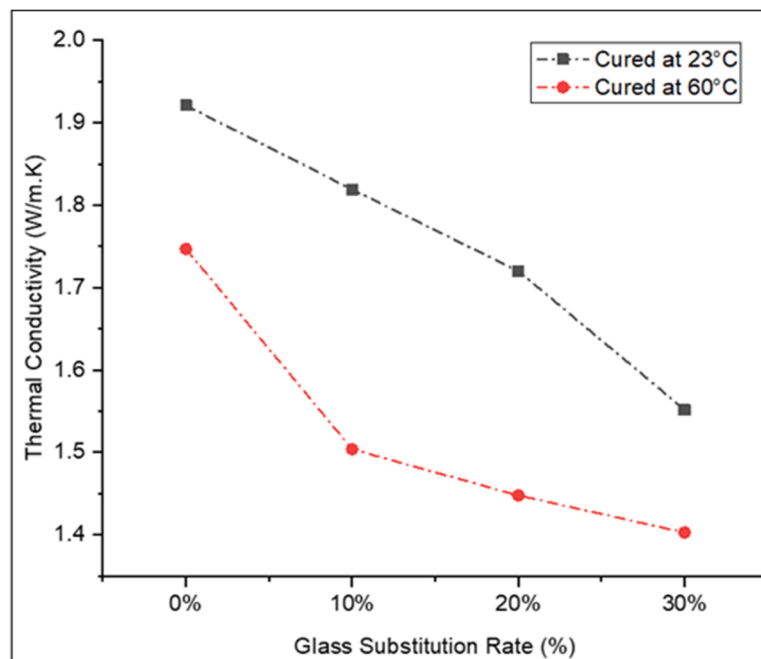


Fig. 14. Thermal conductivity of geopolymer mortars at two curing temperatures (23°C and 60°C).

accelerated reaction mechanisms, respectively. The samples were then directly transferred to a ventilated oven at ambient temperature to simulate a prolonged moisture-loss environment. The outcomes of this study showed that the initial preparation temperature slowed the reaction kinetics, thereby disrupting the usual flow of dissolution and polycondensation steps. This is followed by extensive moisture loss, which has affected the overall gel formed. This results in a heterogeneous microstructure regardless of the curing temperature used for 24 hours, as well as a decrease in compressive strength. Normally, curing for 24 hours at 60°C accelerates dissolution at an early stage and facilitates rapid gel formation. This temperature was selected for comparison with the standard behavior of geopolymers at 60°C. However, even at

elevated temperatures, if the dissolution of reactive species is hindered and delayed, subsequent gel formation will require additional time to rearrange and establish a robust cross-linked gel network. Moreover, when this process is still in its initial, weak structuring phase, the drying condition effectively removes water, an essential component during the first two stages of the reaction. This loss of moisture disrupts the development of cross-linking bonds within the matrix, resulting in a weaker material with high porosity and reduced compressive strength.

4.2. Evolution of pore network

The adopted curing method has directly affected the microstructure

of geopolymers. A slow reaction retards effective gel formation, yet the gel continues to reorganize. However, this reorganization was disrupted again when the material was directly exposed to excessive drying. This sudden moisture loss creates cracks and pores, resulting in a heterogeneous microstructure instead of a dense, well-compacted geopolymer. Microstructural observations support this mechanism, with a large network of pores and cracks increasing as curing days increased. Moreover, quantitative porosity and compressive strength results also confirmed this observation. As curing days increased, porosity increased while compressive strength decreased.

4.3. Effect of pore network on transport properties

It is evident from this research that porosity alone is not the only parameter affecting the mechanical performance of geopolymers; pore and crack connectivity also play a key role. The same microstructural features explain the gas permeability measurements. Gas transport primarily occurs via continuous preferential pathways that connect neighboring pores and cracks. Consequently, isolated pores contribute relatively little to permeability, whereas interconnected defects considerably facilitate fluid transport. The higher permeability measured for the specimens is therefore consistent with microscopy observations showing a less compact, more continuous pore network. Although pore connectivity was not measured directly in this study, the combined microscopy observations and transport measurements strongly suggest that the continuity of the connected crack-pore network is a governing parameter controlling permeability, thermal conductivity, and chloride migration. Once a continuous pore-cracks network develops, chloride ions can migrate more rapidly through the geopolymer matrix under the applied electrical field. Consequently, specimens exhibiting higher permeability also display greater chloride penetration depths, and thus a higher diffusion coefficient. This consistency among microscopy observations, permeability measurements, and chloride migration results demonstrates that these transport properties are governed by the same underlying microstructural parameter, namely, the continuity of the pore and crack network formed during curing. This convergence between independent experimental techniques considerably strengthens the proposed interpretation of the curing mechanism.

4.4. Coupling microstructure, transport properties, and mechanical performance

The progressive evolution of the microstructure ultimately explains the observed mechanical behavior. In well-ambient-cured geopolymers, the continuous amorphous aluminosilicate gel forms an efficient load-bearing skeleton that uniformly transfers applied stresses throughout the material. Conversely, incomplete geopolymerization leaves a weak gel and residual pores that become preferential sites for microcrack initiation. As these defects progressively interconnect, local stress concentrations develop under compressive loading, facilitating crack propagation and premature failure. Therefore, compressive strength should not be interpreted solely as a function of total porosity but rather as the consequence of the overall integrity of the geopolymeric skeleton. The experimental results consistently demonstrate that the combined effects of incomplete gel formation, higher residual porosity, progressive microcrack development, and increasing pore-network continuity collectively control both the mechanical behavior and the transport properties of the investigated materials. Overall, the results from compressive strength testing, porosity measurements, numerical microscopy observations, gas permeability measurements, and chloride migration converge on a unified microstructural mechanism. The curing procedure governs the kinetics of geopolymerization, which controls the development of the amorphous binding gel and ultimately determines the continuity of the pore network. This microstructural evolution explains, in a consistent manner, the variations observed in all

experimental tests, highlighting the critical role of curing in achieving durable geopolymer materials.

5. Conclusion

This research study emphasizes the importance of exploring meta-kaolin/glass powder based geopolymer systems under low-temperature preparation conditions (10-15°C), which better reflect practical applications and substantially influence reaction mechanisms and material performance. The results indicate that reducing the temperature without appropriate mix design and curing alterations may hinder the geopolymerization reaction and, by that, affecting its microstructure, mechanical and transfer properties. The key findings of this research are as follows:

- The combination of low temperature and moisture loss appears to hinder geopolymerization, resulting in a heterogeneous microstructure characterized by inter-connected pores and microcracks, and incomplete reaction products. Consequently, it influences the transport, mechanical, thermal, and durability properties of the geopolymer.
- A 24 hour initial curing at 60°C was insufficient to provide a sustained improvement if it is initially slow and maintained under drying conditions.
- Although glass powder is predominantly highly amorphous and rich in reactive silica and calcium content, its impact remained limited in the investigated conditions. Also, the delayed reorganization and precipitation of Ca- and Si-rich phases within the pores lead to local matrix densification that remains mechanically brittle, and neither stops transport flow nor enhances the mechanical and durability characteristics of the geopolymer material.
- Calcium content derived from the glass powder was not sufficiently adequate to enhance the reaction at least by day 180, as the geopolymer remains to reorganize its gel and overall internal and external structure.
- The low manufacturing temperature as well as the curing (drying) conditions affect the development of the geopolymer microstructure, increasing the pore and crack network connectivity and directly affecting the long-term mechanical, transport, and durability performance.
- These research outcomes highlight the importance of curing conditions and the necessity of optimizing geopolymer for low-temperature applications, given proper mix proportions and the selection of raw materials for that condition.
- Further investigation is needed into this dissolution-controlled non-classical reaction and the effects of changing drying conditions. It would also be valuable to explore whether extended curing at room temperature could improve the reaction prior to drying conditions. Notably, assessing fresh properties, mechanical strength, and durability, such as acid resistance and freeze-thaw resistance, could provide deeper insights into the material's performance both in its initial state and after later curing.

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CRediT authorship contribution statement

Carina Frangieh: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Marianne Saba:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding

acquisition, Formal analysis, Data curation, Conceptualization. **Alexandre Godin**: Writing – review & editing, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Marie Duquesne**: Writing – review & editing, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ameur El Amine Hamami**: Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare no conflicts of interest.

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Data availability

Data will be made available on request.

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