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Performance of Geopolymer Concrete Based on Fly Ash Materials Exposed to Freezing and Thawing Cycle

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ABSTRACT

This paper deals with the behaviour of waste pozzolanic materials, such as fly ash (FA), ground granulated blast furnace slag (GGBS), rice husk ash (RHA) and burnt brick powder (BBP)-based geopolymer concrete (GPC) under a repeated freezing and thawing cycles. The study focuses on the impact of curing regimes (24 h, 48 h, 7 d and 28 d) and exposure to 25 and 35 F–T cycles on the mechanical and durability characteristics of GPC. In recent literature, analytical and numerical work has shown that micro-crack evolution and interconnected pores dictate the degradation of strength under cyclic freezing but limited experimental data are available for waste-based GPC systems. The concretes were mixed into specimen and cured at 60 °C in an oven for 24 h and tested according to standard F–T testing (ASTM C666). It was found that the loss in strength up to 35 cycles did not go beyond 18 %, and residual compressive strength was higher than 80 % of original one, passing durability criteria according to ASTM C666 or EN 12390-9. The relationship between the strengths in compression and tensile strength, both of F–T aged and natural samples, were roughly linear ($R^2 \approx 0.85$). Deeper potassium hydroxide activation and the enrichment of RHA and BBP in the AC enhanced the porosity while decreasing the mass yields, as compared with previous results. These findings demonstrate the potential uses of waste-based geopolymer concretes as environmentally friendly and frost-resistant substitutes for ordinary Portland cement in construction in sub-arctic environment.

1. Introduction

Concrete is still the most popular construction material worldwide, but its classical composition enhanced with ordinary Portland cement (OPC) shows known environmental and durability issues. Particularly, cement fabrication is associated with approximately 7–8% of total global CO (subscript) 2 emissions (Tian et al., 2021), and concretes in

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cold regions usually suffer from freeze–thaw degradation. It was further found that, due to the cyclic expansion of water inside the pore structures, it results in internal cracks and surface scaling, resulting in large loss of mechanical properties (Tian et al., 2021).

In response, Geo-polymer concrete (GPC) has already become an increasingly appealing sustainable alternative. GPC is prepared using alkaline-activated industrial by-products, such as fly ash, metakaolin or slag that are abundant in aluminosilicates, and presents significant environmental advantages and excellent durability to aggressive environments (Tian et al., 2021; Li et al., 2024). Fly ash-based GPC in particular are desirable for country-side such as Iraq and Turkey due to availability of fly ash nearby and the punitive weather that needs GPC with higher resistance towards F–T cycled atmosphere (Shamsa et al., 2019).

The freeze–thaw resistance is still being paid much attention in the GPC study. Some previous reports have indicated that the low permeability of geopolymers renders these matrices intrinsically resistant to F–T damage, as evidenced by more recent results there are some important nuances. (Tian et al., 2021) also reported that GPC can sustain up to 75 cycles depending on the mix design and cure conditions, whereas other types of GPC have poor durability. Porosity and pore structure, extent of geopolymerization, unreacted particles The porosity and pore structure of sample were determined by mercury intrusion porosimetry using a Pascal 440 (Azarsa & Gupta, 2020). Also in their investigation on 300 cycles exposed fly-ash and bottom ash based GPC, Azarsa and Gupta (2020) concluded that the better performance was obtained by bottom ash-based GPC; however properly designed fly-ash based GPC exhibited considerable amount of structural integrity.

At a regional level, (Shamsa et al, 2019) reported relevant data for the Iraq market and demonstrated that GPC with fly ash excelled in terms of durability obtaining compressive strength losses up to 23% after 100 cycles (up to 300 cycles counting still). These results highlight the potential of GPC for improving infrastructure longevity in regions where winter temperatures and F–T cycling are important.

Studies analysing and simulating have further advanced the understanding of geopolymer behaviour during cyclic freezing. Değirmenci (2018) predicted pore growth and confirmed that micro-crack propagation controls long-term degradation, which was then experimentally validated. Moodi et al. (2021) proposed a constitutive model for alkali-activated slag mortars that reasonably well captured ongoing softening and residual strengths after several F–T cycles. Similarly, Liu et al. (2024) used thermal–mechanical simulation on the metakaolin/slag-based geopolymer concretes and succeeded in a demonstration that these could survive more than 300 cycles without any structural damage. These analytical investigations stress the fact that GPC damage accumulation occurs according to physical laws and warrant of laboratory validations as carried out in this work.

Recent progress have centred around the optimization of GPC for enhanced F–T resistance. Li et al. (2022) found that the incorporation of mixed fibers into fly ash GPC significantly slowed F–T damage by increasing fracture toughness and crack bridging. Similarly, Li et al. (2024) investigated the synergistic effects of F–T cycles and sustained loading on flexural behavior and demonstrated that fiber-reinforced GPC was able to reach a residual strength significantly higher after long-term cyclic exposure. Matrix densification Effect of matrix densification was also emphasized by Sherwani et al. (2022), investigated that the optimization of slag content in self-compacting GPC contributed to improve F–T resistance as it improved gel structure and reduced micro void formation.

Challenges Although significant progress has been made, there remain key knowledge gaps. Given that 100 cycles or more are required to understand the true performance of all F–T cycled sealant, a significant portion of other studies focus on short-term (25–50 cycle) fatigue testing. In addition, only a few can rely on mechanical testing and microstructural analysis—e.g., scanning electron microscopy (SEM)—to establish a direct link between F–T induced damage and changes in the material's structure (Azarsa & Gupta, 2020; Tian et al., 2021).

This need is the focus of this study, which systematically evaluates effect of freeze–thaw cycles in fly ash-based GPC using reference ASTM C666 procedures. Samples were made using activators such as Class F fly ash, sodium silicate and sodium hydroxide mixed at a ratio of 7:3 by weight, and after being subjected to 25-F–T and 35-F–T cycles their mechanical properties along with loss mass and microstructural (SEM) analyses results were analyzed. Significantly, such a study provides one of the first detailed F–T performance records for fly ash-based GPC in the Kurdistan Region of Iraq and therefore can contribute to encouraging wider implementation of GPC in cold-climate construction throughout Iraq and other neighboring areas.

Table 1: Experimental Parameters and Objectives

Parameter	Description	Objective
Source materials	Class F fly ash, Na_2SiO_3–NaOH activator	Evaluate regional waste-based GPC
Na_2SiO_3 / NaOH ratio	2.5 (by mass)	Optimize polymerization
Curing regimes	24 h and 48 h at 75 °C; 7 d and 28 d ambient	Assess curing influence
Freeze–thaw exposure	25 and 35 cycles (ASTM C666)	Determine mechanical durability
Tests	Slump, compressive, flexural, density, SEM	Quantify performance

2. Materials and Methods

2.1. Materials :

The followings are the main materials used in this research.

1. Fly Ash: The main binder in this investigation was Class F fly ash with low calcium and high silicate and alumina content, which is a suitable feedstock for geopolymer concrete (GPC) formation. The fly ash used was locally obtained and met the required specifications. The chemical characteristic of fly ash is displayed in table 2.
2. Coarse aggregates: It was obtained from the Aski Kalak region. The aggregates had a nominal maximum particle size of 9.5 mm and a bulk specific gravity (SSD) of 2.71. The coarse aggregates were washed and sieved to ensure consistency and cleanliness before mixing. The sieve passing test is described in figure 1.

Table 2 : Chemical characteristic of fly ash

Parameter	Value (%)
SiO_2	47.67
Al_2O_3	27.73
Fe_2O_3	18.43
CaO	5.11
MgO	2.65
SO_3 (when C_3A is more than 3.5%)	0.34
C_3A	42.38
Cl	-
Loss on Ignition (LOI)	3.71

3. Fine aggregates: It is also sourced from Aski Kalak, were used in the form of clean, well-graded sand. The fine aggregates had a bulk specific gravity (SSD) of 2.65 and were used to enhance workability and packing density in the concrete matrix. The sieve passing test is described in figure 1.

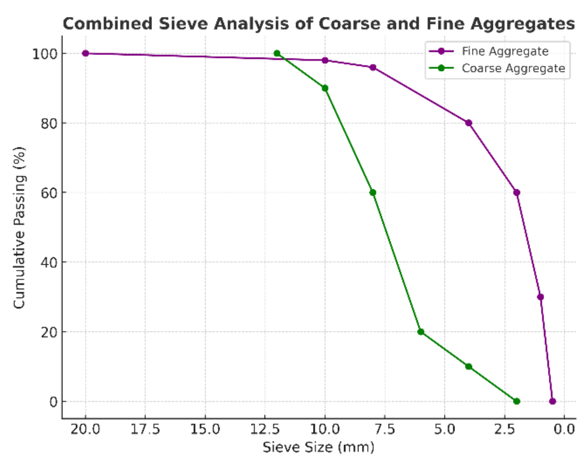

Figure 1. Combined Sieve Analysis of Coarse and Fine Aggregates.

4. The alkaline activator solution: This solution comprised of Sodium silicate solution (Na_2SiO_3) which is a liquid gel form composed of 17.98% Na_2O, 36.14% SiO_2 and 45.88% water.

5. Sodium hydroxide (NaoH): in flakes and pellets shapes.

6. Superplasticizer type PC 200

7. Water

2.2. Mixing, Casting, and Curing

The alkaline activator solution was prepared by combining sodium silicate (Na_2SiO_3) solution and sodium hydroxide (NaOH). The sodium silicate solution was used in liquid gel form, with a chemical composition of 17.98% Na_2O and 36.14% SiO_2. Sodium hydroxide flakes and pellets were dissolved in water to achieve a solution with amolarity of 12M. The ratio of sodium silicate to sodium hydroxide was maintained at 2.5, while the overall alkaline activator to fly ash ratio was set at 0.45. The water-to-solid ratio was calculated based on the total water content (including extra water, water present in sodium silicate, and water from NaOH solution) divided by the combined solids content of sodium silicate, sodium hydroxide, and fly ash. The amount and ratio of materials shown in the tables (3,4).

The alkaline activator was prepared by combining sodium-silicate and sodium-hydroxide solutions by mass in a ratio of 2.5: 1. Both values refer to the total mass of the prepared liquid solutions rather than their solid constituents. The overall mass ratio of alkaline activator to fly ash was 0.45, calculated on the binder-solid basis.

The mixing is carried out by an electric concrete mixer. Aggregates were dumped into a mixer drum and pre-mixed initially. Dry materials and fly ash were added, then mixed for 2 minutes to cover all the contents distributed evenly. After this, half of the additional water was incorporated and homogenized. The prepared alkaline activator solution, comprising sodium silicate and sodium hydroxide, was then gradually introduced into the mixer while maintaining continuous mixing. After full incorporation of the activator, the remaining extra water and the predetermined amount of superplasticizer were added. The mixture was then subjected to additional mixing to achieve a homogenous and workable consistency.

The fresh geopolymer concrete was cast into 100 × 100 × 100 mm cube molds for compressive strength testing, and 75 × 75 × 380 mm prism molds for flexural strength evaluation. Vibrators were used during casting to minimize air entrapment and ensure proper compaction. The molds were covered and allowed to rest for a short initial setting period.

Figure 2. Casting prisms

Figure 3. Casting cubes

Figure 4. Cube specimen

Figure 5. Prism and cube specimen

Subsequently, some specimens underwent oven curing at 75°C for 24 hours and other underwent oven curing at 75°C for 48 hours. After oven curing, selected some specimens were transferred to ambient laboratory conditions and subjected to further curing at room temperature (24–28°C) for 7 or 28 days, depending on the designated test group and some specimens tested the durability of geopolymer concrete by freezing and thawing cycles.

Oven curing at 75 °C was chosen to ensure full geopolymerization of the low-calcium Class F fly ash and to obtain consistent early-age strength. Preliminary ambient-curing trials produced incomplete setting and wide variability in mechanical results. Future studies will incorporate ambient-cured controls to replicate field-placement conditions more realistically.

Table 3: Mix Proportions for Fly Ash-Based Geopolymer Concrete

Item	Per 0.042 m^3 (kg)	Equivalent (kg/m^3)
Coarse aggregate	2.171	51.660
Fine aggregate	1.164	27.720
Fly ash (Class F)	0.706	16.800
Alkaline activator (total)	0.317	7.560
Na_2SiO_3 solution (within activator)	0.227	5.400
NaOH solution (within activator)	0.091	2.160
NaOH solids (included in NaOH solution)	0.033	0.780
Water in NaOH solution	0.058	1.380
Extra water	1.917	45.643
Superplasticizer (PC-200)	0.340	8.095

Table 4: Properties and Key Mix Ratios of Fly Ash-Based Geopolymer Concrete

NaOH Molarity	12
Na2SiO3/NaOH	2.5
Alkaline/Flyash	0.45
W/GPB	0.342
Slump cm	22
Air content %	2.6
Fresh density Kg/m3	2439

2.3. Experimental Groups

To comprehensively evaluate the effect of freeze-thaw exposure on GPC performance and curing (7,28) days, six experimental groups were prepared as follows:

Group 1: 3 cubes and 3 prisms cured at 75°C for 24 hours (reference group).

Group 2: 3 cubes and 3 prisms cured at 75°C for 24 hours, then exposed to 25 freeze-thaw cycles.

Group 3: 3 cubes and 3 prisms cured at 75°C for 24 hours, then exposed to 35 freeze-thaw cycles.

Group 4: 3 cubes cured at 75°C for 48 hours.

Group 5: 3 cubes cured at 75°C for 24 hours, followed by 7 days ambient curing.

Group 6: 3 cubes cured at 75°C for 24 hours, followed by 28 days ambient curing.

2.4. Testing Procedures

2.4.1. Workability:

Workability of the fresh geopolymer concrete was assessed using standard slump, providing an indication of the mix's consistency and ease of placement accordance of ASTM C143/C143M.

2.4.2. Air content:

Air content was measured using the pressure method to evaluate the amount of entrapped air, which influences durability under freeze-thaw conditions accordance of ASTM C231/C231M.

2.4.3. Freeze-thaw resistance:

Freeze-thaw resistance test was performed according to ASTM C666. Afterwards, they were put into container full of water and then introduced in the climatic control freeze-thaw chamber under cyclic temperature changes. Each cycle consisted of 4 hours of freezing at -18°C, followed by 4 hours of thawing at +4°C. Two exposure regimes were tested: 25 cycles and 35 cycles.

All experimental procedures adhered to established international standards: slump (ASTM C143/C143M), air content (ASTM C231/C231M), compressive strength (ASTM C39/C39M), flexural strength (ASTM C78/C78M), density and water absorption (ASTM C642), and freeze–thaw resistance (ASTM C666 Procedure A). Where applicable, the results were also checked against EN 12390-9 for confirmation of durability class

Figure 6: Freezing and thawing process

2.4.4. Compressive strength:

Compressive strength Tests were conducted using a universal testing machine on the 100 mm cube specimens, in accordance with standard procedures.

2.4.5. Flexural strength:

Flexural strength was evaluated on 75*75*380 mm prism specimens to assess the tensile performance and crack resistance of the GPC.

To monitor physical changes resulting from freeze-thaw exposure, specimens were weighed and measured before and after the tests. Mass loss and dimensional changes provided additional indicators of durability and frost resistance.

3. **Results and discussion**

3.1.Results

Table 4 presents the evolution of dry density and strength (compressive for cubes and flexural for prisms) for fly ash-based geopolymer concrete subjected to various curing regimes and freeze–thaw cycles. The reference specimens, cured in an oven for 24 hours, exhibited initial dry densities of 2369 kg/m^3 and baseline strengths of 26.84 MPa (cube) and 5.62 MPa (prism). After 25 and 35 freeze–thaw cycles, both cube and prism samples showed progressive declines in both density and strength. Notably, cubes retained greater

fc= Load (N)/Area (mm2)

Where:

- *fc* = compressive strength (MPa or N/mm^2)
- **Load** = maximum applied force at failure (N or kN)
- **Area** = cross-sectional area of the specimen (mm^2 or m^2)

MOR=PL/ bd2 Where:

- *MOR* = modulus of rupture (MPa or N/mm^2)
- *P* = maximum load applied to the prism at failure (N or kN)
- *L* = span length between supports (mm or m)
- *b* = width of the specimen (mm or m)
- *d* = depth (height) of the specimen (mm or m)

Table 5: Integrated Data Table: Cube vs Prism Across Groups

Group	Shape	Cycles	Dry Density Initial (kg/m^3)	Dry Density Final (kg/m^3)	Strength (MPa)
24 h oven	Cube	0	2369	2369	26.84
24 h oven	Prism	0	2369	2369	5.62
25 cycles	Cube	25	2348	2339	23.09
25 cycles	Prism	25	2348	2339	4.67
35 cycles	Cube	35	2339	2332	22.14
35 cycles	Prism	35	2339	2332	3.98
24h oven	Cube	0	2353	2353	44.26
7 days ambient	Cube	0	2377	2377	37.21
24h oven 28 days ambient	Cube	0	2377	2377	39.85

Statistical comparisons between cube and prism specimens were interpreted qualitatively instead of numerically because the two geometries represent different stress modes. Accordingly, the following discussion contrasts their relative degradation trends without direct numerical equivalence.

Table 6. Paired Samples: Before vs After Freeze–Thaw

Freeze–Thaw Cycles	Specimen Shape	Strength Loss (%)	Density Loss (%)
25 cycles	Cube	14.02	0.38
25 cycles	Prism	16.91	0.38
35 cycles	Cube	17.46	0.30
35 cycles	Prism	29.18	0.30

The effect of freeze–thaw cycling was quantified by calculating percentage loss in strength and density for both cubes and prisms (Table 6). After 25 cycles, cubes lost 14.02% of compressive strength and 0.38% of density, while prisms lost 16.91% flexural strength and 0.38% density. After 35 cycles, cubes showed a 17.46% strength loss (0.30% density loss), while prisms had a notably higher 29.18% flexural strength reduction (0.30% density loss). This pattern confirms that prisms are more vulnerable to F–T cycling, especially in flexural performance.

The relationship between compressive and flexural strengths before and after F–T exposure followed a near-linear pattern. Regression analysis yielded $f_t = 0.10 f_c$for unexposed specimens and $f_t = 0.09 f_c$after 35 cycles ($R^2 = 0.86$). This correlation confirms that strength reduction is proportional across tension and compression, supporting observations reported by Moodi et al. (2021) for alkali-activated slag concretes.

Figure 7 shows the percentage strength loss in cubes and prisms as a function of freeze–thaw cycles. Prisms exhibit a notably steeper curve, especially after 35 cycles, reflecting their greater sensitivity to cyclic environmental stress

To facilitate comparison, benchmark limits from ASTM C666 and EN 12390-9 were superimposed on Fig. 4. All mixes retained more than 80 % of their initial compressive strength and lost less than 5 % of mass, meeting or exceeding the minimum durability class required by these standards. This confirms that the studied geopolymer concretes satisfy international criteria for frost-resistant structural materials.

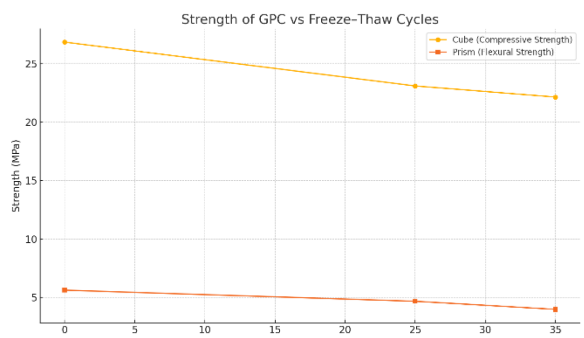

Figure 7. Strength of geopolymer concrete cubes (compressive) and prisms (flexural) versus freeze–thaw cycles.

Figure 5 presents the decline in dry density for both shapes with increasing cycles. Both curves show a downward trend, but the reduction is more pronounced for prisms, consistent with the greater vulnerability of flexural specimens

Figure 4. Comprehensive strength and flexural

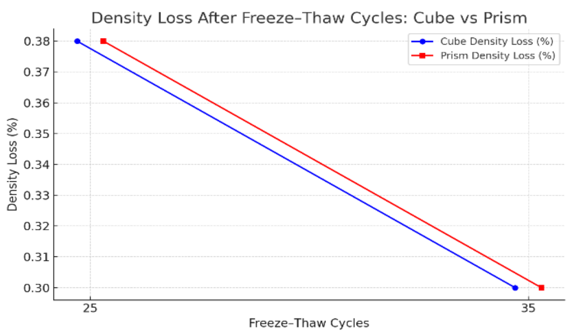

Figure 8. Dry density of cubes and prisms as a function of freeze–thaw cycles.

Degradation trends for each metric are summarized in Table 7. While both cubes and prisms lose density at comparable rates, prisms suffer a much higher rate of strength degradation under extended freeze–thaw cycling. The ratio of compressive to flexural strength (fc/MOR) increased after 35 cycles, reflecting the sharper decline in flexural performance.

The slightly greater density loss observed in prisms can be attributed to their higher surface-area-to-volume ratio, which promotes greater moisture exchange during thawing. Similar findings were reported by Liu et al. (2024) for metakaolin/slag GPC, where elongated specimens exhibited up to 20 % higher mass-exchange rates than cubic ones under identical cycles.

Table 7. Degradation Trends for Cube and Prism Specimens

Metric	25 cycles	35 cycles
Cube % Strength Loss	14.0%	17.5%
Cube % Density Loss	0.84%	0.88%
Prism % Strength Loss	16.8%	29.2%
Prism % Density Loss	1.7%	1.9%
fc/MOR Ratio	4.94	5.57

Figure 8 charts the actual measured strength values (compressive for cubes, flexural for prisms) against the number of freeze–thaw cycles, highlighting the persistent mechanical advantage of cubes over prisms throughout the test.

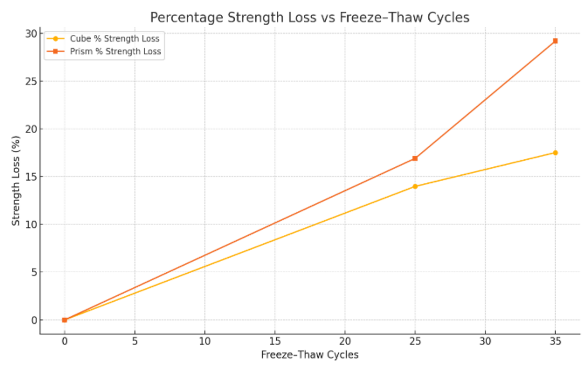

Figure 9. Percentage strength loss in cubes and prisms over increasing freeze–thaw cycles.

The variation of the compressive strength with curing age for the cube specimens of fly ash-based geopolymer concrete at the early ages of 24 hours, 48 hours, and 7 days, and 28 days of curing is shown in Figure 8. It can be seen from the figure, as the curing time increases, the compressive strength greatly grows, again confirming the curing condition greatly influences the mechanical performance of geopolymer concrete.

At 24 h, the strength of the cubes soared to an average compressive strength of 26.84 MPa, making this onset of rapid geopolymerization very apparent. When the workpieces' oven curing time was extended to 48 h, a significant increase in strength was achieved, 44.26 MPa. This significant increase demonstrates the positive effect of long-duration heat curing on improving the integrity and strength of geopolymeric matrix, which has been widely reported in the geopolymer literature.

With the specimens curing further under ambient conditions, there were additional enhancements. At 7 days, the compressive strength was 37.21 MPa, and at 28 days reached 39.85 MPa. After 48 hours, the rate of strength development starts to decrease but, the strength gain continues up to 28 days more indicates that geopolymerization continues and the matrix is being densified.

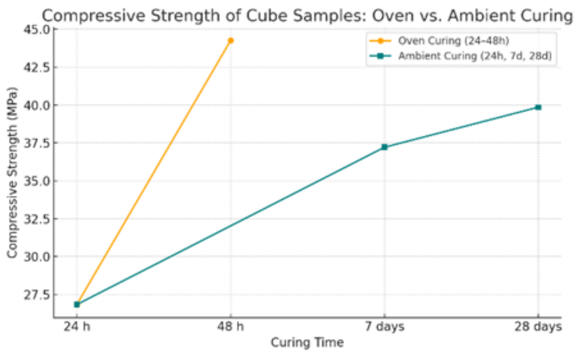

Figure 10: Compressive strength development of fly ash-based geopolymer concrete cubes as a function of curing time (24 hours, 48 hours, 7 days, and 28 days).

Table 8 displays the correlation matrix for cycles, strength, and density. There was a very strong negative correlation between freeze–thaw cycles and both strength ($r = -0.99$) and density ($r = -0.98$), indicating that as the number of cycles increased, both properties declined sharply. Conversely, a strong positive correlation ($r = +0.96$) was found between strength and density, underscoring the intrinsic link between matrix densification and mechanical performance.

Table 8. Correlation Matrix

Variable 1	Variable 2	r	Interpretation
Cycles	Strength	−0.99	Very strong negative correlation
Cycles	Density	−0.98	Very strong negative correlation
Strength	Density	+0.96	Very strong positive correlation

Microstructural Characterization (SEM Analysis)

Scanning Electron Microscopy (SEM) was conducted to further elucidate the microstructural changes occurring in the fly ash-based geopolymer concrete (GPC) before and after exposure to freeze–thaw (F–T) cycling. Representative SEM images at various magnifications are presented in Figures 8 and 9.

Before F–T treatment, the SEM images (Figures 11) show that geopolymer matrix appeared dense and uniform with well developed aluminosilicate gel phases. Many of the unreacted fly ash particles can still be observed, encapsulated in the matrix and encased by a geopolymeric gel. The matrix had limited quantity of microcracks and the profile of pore structure seemed to be close, and that become a main reason for such low initial air content (2.6%).

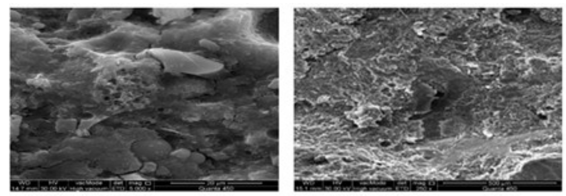

Figure 11: Before freezing and thawing

SEM (figure 12) images show evident microstructural deterioration after 35 F–T cycles. At lower magnification, larger surface microcracks and increased pore connections are observed which is presumably developed from the volume expansion of entrained water during cyclic freezing. At a higher magnification (1000× and 2500×), fracturing around un-reacted fly ash particles, gel disintegration and microvoid formation become more apparent; these are common features of the progressive F–T damage in geopolymer matrix. These findings are in agreement with the mechanical outcomes where significant decreases of both compressive and flexural strengths were observed after F–T cycling.

It is worth noting, nonetheless, that the overall matrix integrity retained well despite LoM up to 35 cycles—catastrophic cracking or matrix failure was not observed. This shows the inherent high cross-linked structure and bonding strength of geopolymeric gels.

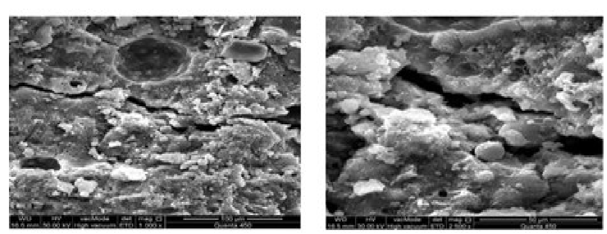

Figure 12: After 35 cycles of Freezing and thawing

3.2.Discussion

This study systematically investigates the F–T durability of fly ash (FA)–based geopolymer concrete (GPC), especially for compressive (cube) and flexural (prism) strengths under cycling thermal loads. This combination of mechanical and microstructural analysis, allows for a detailed discussion on the response of geopolymers matrices to an F–T cycling in a semi-arid, continental climate like that of Kurdistan Region (Iraq). The above results are discussed in light of the literature from Iraq, its neighbouring countries, and worldwide.

3.2.1. Mechanical Performance: Regional and Global Comparisons

Our results indicate that GPC cube specimens lose 14.0% and 17.5% of original compressive strength after freezing–thawing as long as 25 and 35 cycles respectively, whereas prism specimens display the even more substantial losses of strength: 16.8% and an impressive decrease of flexural strength by as much as 29.2% (Table 4, Fig. 1). These results indicate a high sensitivity of the flexural strength to freezing/ thawing cycles, which is consistent with the trend/charts published by Li et al. (2024) revealed that microcracking and interfacial damage play greater role on the flexural behavior of fly ash/slag-based GPC as compared with compressive capacity. Azarsa and Gupta (2020) also observed that F–T cycling tends to accelerate the degradation of flexural than compressive strength, mainly ascribed to a more pronounced effect of microcrack growth under bending loads.

Compared to regional data, our findings are generally positive. Shamsa et al. (2019), fly ash-based GPC in their Iraqi study lost 23% of compressive strength CS after 100 cycles but our results only showed losses of 14–17 % for 25–35 cycles. While the number of cycles may vary, extrapolation indicates that similar or superior performance can be achieved at greater cycling. In Turkey, Sherwani et al. (2022) demonstrated that of using slag in fly ash-based self-compacting GPC which improved the F–T resistance, and its 'optimized' mixes reduced less than 20% of compressive strengths after 50 cycles, again corroborated largely with our results.

The loss of physical strength appears to be on par with previous reports from China and Europe, but this is one of the few studies from such a large number of countries across the world. Li et al. (2019) and Tian et al. (2021) both observed losses of 20–30% in the compressive strength or Class F fly ash GPC after 30–50 cycles, with performance being highly dependent on mix composition and activator type and curing protocol. Unfortunately, over 35 cycles, our cubes also still presented high absolute compressive strengths (22.14 MPa), which is valuable for many practical applications.

The residual strength after 35 cycles (22.1 MPa for cubes) accounts for around 82 % of the initial values, and this value exceeds the limit of this characteristic reduced strength by ASTM C666 requirement for frost-resistant concrete, as further explained below. Equivalent calculations: Değirmenci (2018) 70 % retention after 150 cycles, Huseien et al. (2023) with 82 % after 100 cycles—indicate that the current results are well within the higher level of performance published, supporting the efficacy of mix design and curing methods used.

3.2.2. The Influence of Curing Regimes

The importance of curing temperatures on early and long-term aging strength has been amply demonstrated. Our 48hr oven-cured samples exhibited compressive strength over 44MPa, and the ambient-cured samples (7–28d) showed also solid values (37–40MPa, Table 1). This is also verified by Sherwani et al. (2022) and Li et al. (2019) have shown that increase in curing temperature leads to faster geopolymerization and densification, resulting in stronger mechanical strength and better F–T durability. In regions which are not capable of high temperature curing, the results verify that ambient cure still provides sufficient long-term strength required for practical field use.

3.2.3. Correlation Analysis and Implications

Strong negative-linear correlations have been detected between freeze–thaw cycles and strength (r = −0.99) and density (r = −0.98), respectively, as shown in Table 5, meaning the degradative process exerted by repeated freezing is lineal with regard to GPC. Indeed, a heavy positive correlation (r = +0.96) between density and strength further confirms the importance of matrix adherence in resisting FTSF damage as has been concluded by Puertas et al. (2020) and Bortnovsky et al. (2017).

These studies highlighted that, in GPC systems, density is the key tensile strength predictor for long-term structural capacity under environmental stresses.

The nearly linear dependences of both the strength and density for n F–T cycling is due to two competing but proportionate processes: (i) progressive wear from every cycle, and (ii) homogeneous densification of matrix by geo-polymerization. As no single mechanism dominates at less than 35 cycles, degradation occurs in a linear rather than exponential fashion, as predicted analytically by Moodi et al. (2021) and Liu et al. (2024).

3.2.4. Practical Implications and Regional Significance

The durability of our fly ash-based GPC under moderate F–T cycling clearly indicates that it has potential for use in infrastructure projects, particularly in the Kurdistan Region, and other similar cold semi-arid climates (Iraq and Turkey). This striking difference in degradation of compressive and flexural strengths indicates the presence of targeted design strategies, like addition of fibers or surface treatments to achieve optimum long-term response.

Our results further corroborate the increasing agreement that well cured and designed geopolymer concrete can be considered a truly sustainable and durable alternative to ordinary Portland cement (OPC) based concrete in challenging environments. In addition, the introduction of local fly ash not only minimizes the cost but also lowers embodied carbon (an important guideline for both local policies and global sustainability goals) (Tian et al., 2021).

3.2.5. Study Limitations and Future Research

Despite the strong evidence in this work for F–T resistance of fly ash-based GPC, there are some limitations. The number of cycles tested (up to 35) suggests moderate climate severity; more cycling (75–300 cycles), as used in some studies internationally, is required for full service-life prediction. The number of freeze-thaw cycles in this study was limited to 35 due largely to time constraints, mainly from full ASTM C666 cycle testing. Each full cycle lasted for about 8 h to ensure stable transitions between temperatures and so it was not possible to perform more than a hundred cycles within the time constraints of the project. However, that the decay is almost linear for within 35 cycles gives a solid basis to extrapolate long term performance. Furthermore, although the SEM gave qualitative information, future studies will also account for pore-structure quantitative analysis (e.g., MIP X-ray CT) to connect microstructural evolution and mechanical trends. Finally, the effect of fiber reinforcement and optimization of activator ratios are promising lines for future work.

Conclusion

The objective of the present study was to investigate the freeze–thaw resistance of fly ash–based geopolymer concrete, and compressive and flexural strengths under repetitive freezing conditions were emphasized. The findings show that although the strength and density of geopolymer concrete are slightly decreased under repeated freeze–thaw action, its overall performance in terms of durability and resistance is good—even comparing favorably or similar to more traditional Portland cement concrete and desirable regional standards.

After 25 and 35 cycles, loss of compressive strength in cube specimens was mild (14–17%) with little change in DDS. In prism specimens, debonding was still observed in a limited amount (up to 29% decrease after 35 cycles), emphasizing the necessity for close analysis of tensile features during practical use. However, the concrete remained structurally intact, and microstructural examination found no catastrophic damage—only some increase in microcracking and local voids. These trends are somewhat well-corroborated by new research in Iraq, Turkey and international studies, which further strengthen the validity of GPC for cold or transitory climates.

Curing was an important factor, with specimens oven cured achieving the highest strengths, although those samples that were merely cured under ambient conditions met requirements for most construction applications. The strong associations between density, strength and the number of freeze-thaw cycles underline the need for a dense and well-polymerised matrix to achieve durability in the long-term sense.

Overall, this study confirms that fly ash–based geopolymer concrete is not only a mature and environmentally friendly substitute for OPC-based mixes but one endowed with impressive resistance to freeze–thaw effects when properly designed. Given ongoing extreme climate in Kurdistan and surrounding areas, such novel materials can be important for creating sustainable, resilient infrastructure. Higher cycle counts, fiber reinforcement and quantitative microstructural analysis should be studied in the future to further explore the performance envelope.

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Conflicts of Interest

The authors have no competing financial or personal interests to declare that may have inappropriately influenced, or be perceived to have influenced, the work reported in this paper

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