Cleaner Engineering and Technology 24 (2025) 100853

Contents lists available at ScienceDirect

Cleaner Engineering and Technology

journal homepage: www.sciencedirect.com/journal/cleaner-engineering-and-technology

# Investigating the synergistic impact of freeze-thaw cycles and deicing salts on the properties of cementitious composites incorporating natural fibers and fly ash

Ildiko Merta [a,*], Vesna Zalar Serjun [b], Alenka Mauko Pranjić [b], Aljoša Šajna [b], Mateja Štefančič [b], Bojan Poletanović [a], Farshad Ameri [a], Ana Mladenović [b]

[a] *TU Wien, Faculty of Civil and Environmental Engineering, Karlsplatz 13, 1040, Vienna, Austria*
[b] *Slovenian National Building and Civil Engineering Institute, Dimičeva ulica 12, 1000, Ljubljana, Slovenia*

A R T I C L E I N F O

*Keywords:*
Cementitious composites
Natural fibers
Freeze-thaw cycles
Mechanical properties
Mass loss
Fly ash

A B S T R A C T

In cold climates, concrete structures confront durability challenges due to harsh conditions. This study evaluates the effects of incorporating natural fibers, such as hemp and flax fibers (at 1 vol%), and partially replacing cement with fly ash (at 25 and 50 wt%) on the properties of cementitious composites subjected to accelerated aging under freeze-thaw cycles and deicing salts.

Findings reveal that natural fibers enhance the freeze-thaw resistance, reducing deterioration (scaling) to 5–8% after 56 cycles. When mortars were subjected to accelerated freeze-thaw cycles, the compressive strength of plain mortar significantly decreased (up to 57%). However, adding natural fibers to the matrix substantially reduced its compressive strength loss. In the case of flexural strength, plain mortars experienced 33% loss, while hemp, flax, and polypropylene fiber mortars showed only 13%, 23%, and 10% losses, respectively. Furthermore, mortars experience a notable enhancement in their energy absorption capacity when reinforced with natural fibers, particularly with hemp fibers (up to 348% higher than plain mortar).

Under harsh conditions, hemp and flax-reinforced mortars, with 25 wt% fly ash replacement, lose the compressive strength significantly however still demonstrate an alternative to synthetic fibers in terms of flexural strength. Even with 25 wt% of fly ash, mortars with natural fiber reinforcement display significantly superior energy absorption capacities compared to plain mortars (up to 48%).

| Notation | | | |
|---|---|---|---|
| FTC | Freeze-thaw cycle | OPC | Ordinary Portland cement |
| PP | Polypropylene | FTS | Freeze-thaw with deicing salts |
| FRC | Fiber-reinforced concrete | SEM | Scanning electron microscopy |
| XRF | X-ray fluorescence | ITZ | Interfacial transition zone |
| XRD | X-ray powder diffraction | C-S-H | Calcium silicate hydrate |
| LOI | Loss on ignition | Na | Sodium |
| Fe | Iron | Et | Ettringite |
| Ca | Calcium | Hc | Hemicarboaluminate |
| Si | Silicon | NaCl | Sodium chloride |
| Al | Aluminium | K | Potassium |
| Mg | Magnesium | Mn | Manganese |

(*continued on next column*)

(*continued*)

| Notation | | | |
|---|---|---|---|
| Na | Sodium | P | Phosphorus |
| SCM | Supplementary cementitious material | MIP | Mercury intrusion porosimetry |
| Ea | Energy absorption | FA | Fly ash |
| M | Plain mortar | C-A-Hs | Calcium aluminate hydrates |
| HM | Hemp-fiber reinforced mortar | FM | Flax-fiber reinforced mortar |
| PPM | PP-fiber reinforced mortar | Mc | Monocarboaluminate |
| 3 PB | Three-point bending | EDS | Energy dispersive spectroscopy |

* Corresponding author.
*E-mail addresses:* ildiko.merta@tuwien.ac.at (I. Merta), vesna.zalar@zag.si (V.Z. Serjun), alenka.mauko@zag.si (A.M. Pranjić), aljosa.sajna@zag.si (A. Šajna), mateja.stefancic@zag.si (M. Štefančič), bojan.poletanovic@tuwien.ac.at (B. Poletanović), farshad.ameri@tuwien.ac.at (F. Ameri), ana.mladenovic@zag.si (A. Mladenović).

https://doi.org/10.1016/j.clet.2024.100853
Received 28 June 2024; Received in revised form 18 November 2024; Accepted 25 November 2024
Available online 6 December 2024

## 1. Introduction

Concrete is undeniably the most widely used and highly demanded building material. However, it does have certain drawbacks, including low tensile strength, susceptibility to shrinkage cracking, limited toughness, and high environmental impact as noted by (Betterman et al., 1995; Wang et al., 1990; Banthia and Sheng, 1996). As a result, enhancing the properties of traditional concrete has remained a prominent area of focus in both academic and engineering circles (Tian et al., 2016; Abedi et al., 2023; Abdalla et al., 2022). Researchers have long been engaged in the pursuit of developing a novel composite construction material that possesses exceptional ductility, resistance to cracking, durability, and lower environmental impact. These objectives have been a continuous subject of exploration and study (Jin et al., 2023; Page et al., 2021; Althoey et al., 2023). Assessing the longevity of buildings has become increasingly important, particularly in severely cold regions, where concrete's resistance to freezing is a crucial determinant of its durability. Consequently, the evaluation of durability has emerged as a significant factor in determining the lifespan of concrete structures (Kothari et al., 2020; Tang et al., 2015; Wang et al., 2022). In freezing environments, when a concrete structure is subjected to low temperatures, the water inside its tiny pores freezes and expands. This expansion creates pressure, causing nearby water to move and build up more pressure. If this pressure is stronger than the concrete's strength, it forms tiny cracks that spread (Zeng et al., 2010). The process of freeze-thaw deterioration initiates when water penetrates the empty spaces within concrete. The leaching of calcium hydroxide because of enlarging capillary pores, a product of Portland cement hydration, creates larger voids for water to occupy, accelerating the deterioration process. Freezing of water or salt solution within the concrete pores can cause significant damage and reduce the overall lifespan (Zheng et al., 2022; Zhang et al., 2017a). Water expansion during freezing, where its volume increases by 9% upon turning into ice, exerts substantial pressure on the surrounding concrete, leading to pressure-induced cracking and scaling (Ramachandran and Feldman, 1996). Various concepts have been put forward to explain this particular type of damage, with the hydraulic pressure (Powers, 1958), osmotic pressure (Powers and Helmuth, 1953), and micro-ice-lens model (Setzer, 2001) being the most significant among them. Recent studies (Bayraktar et al., 2021, 2022; Gencel et al., 2021a; Benli et al., 2017) have explored methods to enhance concrete's mechanical and durability properties under harsh environmental conditions like FTCs, including the effects of treated recycled aggregates, optimized cement dosage, waste marble powder, and silica fume to mitigate degradation.

A notable breakthrough in designing of eco-friendly material research is using natural fibers to reinforce cement-based composites (Merta, 2016; Baheti and Militky, 2016). Although synthetic fibers like steel and Polypropylene (PP) are commonly used to enhance the mechanical properties of cementitious composites, they are expensive and can have adverse effects on the environment (Song et al., 2021). In contrast, natural fibers offer a cost-effective, long-term, and environmentally friendly alternative as they require less energy during manufacturing (Balaguru and Shah, 1992) and are usually agricultural waste.

Incorporating fibers into concrete has been shown to have several beneficial effects. It can prevent and control the formation and progression of micro- or macrocracks (Balaguru and Shah, 1992; Bentur and Mindess, 1990), thereby addressing some of the limitations of concrete (Zhao et al., 2023) and reducing damage in freeze-thaw environments (Dong et al., 2021; Balaguru and Ramakrishnan, 1986). The specific role of fibers in concrete depends on their characteristics, such as type, length, diameter, aspect ratio, and tensile elastic modulus (Afroughsabet et al., 2016). Steel and artificial fibers (polypropylene, polyvinyl alcohol, and carbon) in concrete have been well explored under FTCs (Al Rikabi et al., 2018; Mu et al., 2002; Ding et al., 2018; Karbhari, 2002). Richardson (Richardson et al., 2012) investigated the effect of FTCs on PP fibers in concrete, finding that fiber-reinforced concrete showed better performance under freeze-thaw conditions. However, to foster sustainability in the construction industry, the focus of the research was on natural fibres as a replacement for traditional fibres. Currently, there is limited research on the behavior of natural fibers in concrete under freeze-thaw conditions. Affan and Ali (2022) examined the mechanical properties of jute fiber-reinforced concrete under freeze-thaw conditions and found that the mass loss was higher (up to 1.86%) due to water absorption of jute. They also observed that the enhanced toughness index helped control the void propagation in concrete roads during freeze-thaw cycles. However, according to the authors' best knowledge, there is still a lack of information on the detailed behavior of natural fibre-reinforced composites under FTC, and no information on the physical and mechanical properties of mortars reinforced with hemp or flax fibres.

Natural fibers possess several advantageous characteristics, such as biodegradability, low density, availability, strength, and, above all, sustainability (Bogoeva-Gaceva et al., 2007; Onuaguluchi and Banthia, 2016; Merta et al., 2017). The use of agricultural by-products and recycled waste as renewable resources for sustainable production of high-added value products is one of the main principles of circular economy, which has recently been recognized in Europe as an important part of the EU's Green Deal and Circular Economy ambitions (Fritsche et al., 2020). The key components of natural fibers are cellulose, hemicellulose, and lignin (Chokshi et al., 2022). The mechanical performance of these fibers depends mainly on the cellulose content and microfibril angles (Komuraiah et al., 2014). Ziane et al. (2020) found that concrete reinforced with 0.25% hemp fibers exhibited similar performance to that reinforced with polypropylene fibers in aggressive environments. In the field of green and sustainable construction, significant efforts have been made to develop alternative high-performance engineering materials (Zakaria et al., 2015).

Unfortunately, when natural fiber-reinforced cementitious composites undergo aging (cyclic wetting and drying or freezing and thawing), it can lead to reduced strength and toughness of the material. It has been reported that degradation occurs due to fiber mineralization, caused by the migration of hydration products, primarily $Ca(OH)_2$, to the fiber structure. Various studies have explored this durability issue and proposed solutions to prevent fiber mineralization (Mohr et al., 2005; Tonoli et al., 2007; Tolêdo Romildo et al., 2003). Degradation of natural fibers in cement composites (weakening of their structure) was assigned also to the alkaline pore water dissolving lignin and hemicellulose in the fibers. Additionally, alkaline hydrolysis of cellulose molecules occurs, reducing polymerization and lower tensile strength (Filho et al., 2013; Tolêdo Filho et al., 2000). One effective method is substituting a higher amount of Portland cement with fly ash (FA), reducing the calcium hydroxide content in the matrix. Bayraktar et al. (Gencel et al., 2021b) investigated the properties of foam concrete reinforced with hemp fibers and supplemented with FA, optimizing the composites properties through the Taguchi optimization method. Their research highlights the potential of FA and hemp fibres in enhancing durability and sustainability, especially under freeze-thaw conditions.

Including pozzolans such as FA as a cement replacement in the concrete mix not only contributes to a sustainable built environment, reducing industrial waste and landfill usage but also could reduce the degradation of natural fibers due to the reduction of pH (De Souza Rodrigues et al., 2006). High-quality FA enhances concrete cohesion within the mixture. Additionally, FA aids in the long-term development of higher compressive strengths, making the concrete resistant to freezing forces. Concrete containing up to 40% FA by weight of cement demonstrates durability against de-icing salt scaling, while higher amounts of FA render the concrete non-durable. Regardless of the FA content, concrete with FA performs well under freezing and thawing conditions, mitigating internal damage. In many situations, concrete with FA outperforms concrete mixed with Portland cement alone (Knutsson et al., 2011).

The presence of de-icing agents or seawater worsens damage to concrete surfaces, causing increased spalling, a phenomenon known as frost-salt scaling (Verbeck and Klieger, 1957). The "glue spall" mechanism explains this, detailing damage due to differential thermal contraction between ice and concrete, leading to outer ice layer fractures and concrete surface cracks (Valenza and Scherer, 2006). Factors like ice thickness, transport, osmotic pressure, hydraulic pressure, and freezing rate influence scaling. Studies also connect scaling to cryo-suction, causing swelling of cement paste around aggregates, thin flakes, and enhanced transport due to prolonged ice-free conditions on the surface (Fagerlund, 1976; Jacobsen and Sellevold, 1994; Liu et al., 2013).

The traditional approach to studying concrete durability has involved examining individual factors in separation, despite real-world concrete deterioration being influenced by multiple factors working together (Zhang et al., 2019). Existing studies mainly focus on the effects of freeze-thaw cycles (FTCs) on concrete reinforced with steel (Mu et al., 2002) and artificial fibers (Al Rikabi et al., 2018), but more attention needs to be given to natural fibers like hemp and flax. This paper investigates the impact of natural fibers when the composites were exposed to FTCs combined with de-icing salts. In order to mitigate the degradation of the natural fibers and the environmental impact of the cementitious composites, FA was utilized. It was shown that natural fibers improve composite freeze-thaw with deicing salts (FTS) resistance, prevent spalling, and enhance toughness capacity.

## 2. Experimental program

### 2.1. Materials

The research utilized CEM II/B-M (S-L) 32,5R from Považská cementáreň, Slovakia, which conforms to the BS EN 197–1:2011 (BS-EN197 and -1: Cement Part 1: Composition, 2011) standard. FA from Vipap Videm Krško d.d. company in Krško, Slovenia, was used as a cement partial replacement in the mortar mixes. The FA is a product generated in the process of biomass combustion, along with minor fiber rejects, sludges, and fillers from paper processing. The geometrical and physical properties of the cement and FA are presented in Table 1. The average particle sizes of the cement and FA are 12 μm and 50 μm, respectively. The results showed that more than 90% of the FA particles were smaller than 350 μm, as illustrated in both Table 1 and Fig. 1.

The chemical composition of FA was defined using X-ray fluorescence (XRF) following the EN 196-2 (British Standards Institution, 2013), while phase and microstructural characteristics were defined by X-ray diffraction (XRD) and Scanning electron microscopy coupled with Energy dispersive X-ray spectroscopy (SEM/EDS). According to the XRF analysis, the FA contains significant amounts of Si, Ca, and Al, followed by Fe, Mg, and minor elements such as K, S, P, Na, and Mn as shown in Table 2. The XRD analysis identified quartz, calcite, hematite, magnetite, gehlenite, anhydrite, and lime as the major crystalline mineral phases in the FA, while minor phases include portlandite, brownmillerite, feldspar, and dolomite. The XRD spectra (Fig. 2) showed an elevated background, indicating the presence of an amorphous phase. SEM/EDS analysis revealed that the FA consists of rounded grains with varying porosity and dense spherules (Fig. 3). The phases analyzed through EDS primarily consist of Si and Al, with varying Si/Al ratios. Minority elements like Na, Mg, Ca, and K were also detected. The SEM/EDS analysis further identified charcoal fragments as a minor ash component.

**Table 1**
Physical properties of cement and FA utilized.

| Characteristic | Raw material: Cement | Fly ash |
|---|---|---|
| Particle size distribution (μm)[a] | | |
| $D_{90}$ | 45 | 350 |
| $D_{50}$ | 12 | 50 |
| $D_{10}$ | 1 | 10 |
| Surface area ($m^2$/g)[b] | 1.6 | 10.0 |
| Density (Mg/$m^3$)[c] | 2.8 | 2.6 |

Legend.
[a] Determined by laser diffraction analysis (CILAS 920).
[b] Determined as BET specific surface area (ASAP, 2020).
[c] Determined according to SIST EN 1097-7.

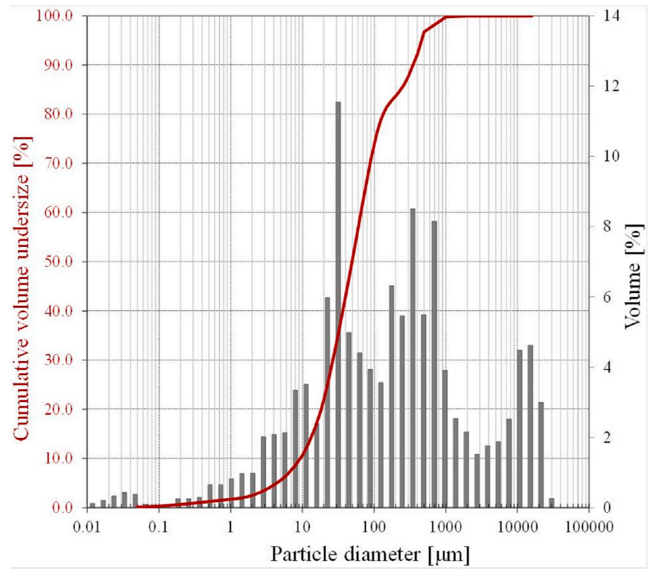


**Fig. 1.** Particle-size distribution of the FA.

To investigate the FA's hydraulic reactivity, FA paste was prepared by blending FA and water in a 1:1 mass ratio. The paste was then cured for several different time intervals, and XRD analyses were conducted at each interval. Fig. 4 confirmed the presence of different Calcium aluminate hydrates (C-A-Hs) after 7 days, specifically Ettringite (Et) and Hemicarboaluminate (Hc), demonstrating the FA's hydraulic activity. As the curing time increased, a transition from Hc to Monocarboaluminate (Mc) was observed.

The mortar mixes were prepared using fine quartz sand as the aggregate (0.4–0.8 mm), along with natural fibers from industrial hemp and flax as reinforcement. The fibers were locally available in Hungary, with diameters ranging from 16 to 50 μm and a length of 10 mm. Synthetic PP fibers were used as a reference material, with a density of 0.91 g/$cm^3$ and tensile strength of 136.5 N/$mm^2$. The hemp and flax fibers had a density of 1.5 g/$cm^3$ and tensile strengths reported in the literature range of 270–900 N/$mm^2$ (Yan et al., 2016). In Fig. 5, the geometry and morphology of hemp and flax fibers under SEM are visible.

### 2.2. Mix design and sample preparation

The mortar mix design was formulated with a precise ratio of binder, sand, and water, specifically at a weight ratio of 1:1:0.4. The water-binder ratio was maintained at 0.4. To reinforce the matrix, fibers were incorporated at a dosage of 1% by volume. To mitigate the influence of the hydrophilic nature of natural fibers, a pre-soaking process was implemented before adding them to the matrix. The fibers were added in a saturated surface dry condition.

The composition details of the mortar mixtures, along with the calculated theoretical densities of the specimens, are comprehensively presented in Table 3. The theoretical density values were determined based on the specific densities of individual constituents present in each mixture. The assumed densities for the respective constituents were as follows: 2.648 g/$cm^3$ for aggregate, 2.800 g/$cm^3$ for cement, 2.580 g/$cm^3$ for FA, 0.997 g/$cm^3$ for water, 1.500 g/$cm^3$ for hemp fibers, 1.500

**Table 2**
Chemical composition analysis of FA.

| Chemical composition (wt.%) | $Fe_2O_3$ | CaO | $SiO_2$ | MgO | $Al_2O_3$ | $SO_3$ | $P_2O_5$ | $Na_2O$ | MnO | $K_2O$ | L.O.I | Total |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Fly ash | 9.37 | 15.18 | 37.02 | 5.48 | 14.85 | 0.86 | 0.42 | 0.41 | 0.20 | 1.49 | 12.26 | 97.54 |

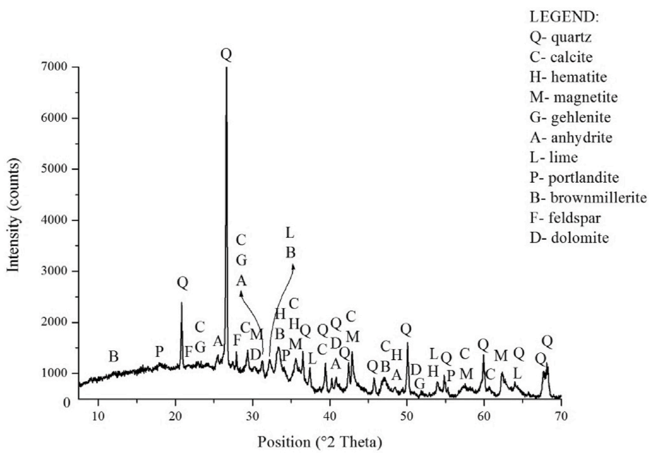


**Fig. 2.** XRD analysis of the FA.

g/cm³ for flax fibers, and 0.9100 g/cm³ for PP fibers.

For the preparation of the mortar, a laboratory drum mixer was employed to ensure consistent mixing. From each mixture, a total of six prism specimens, with dimensions of 40 × 40 × 160 mm³, were cast. After a curing period of 24 h, the specimens were de-molded and subsequently immersed in water until the designated testing age of 28 days for unaged specimens or continued with an accelerated aging technique (for aged specimens).

## 2.3. Methods

In this study, Three-point bending (3 PB) tests were conducted on all samples, using six prismatic specimens with dimensions of 40 x 40 × 160 mm³, following standard EN 1015-11 (European Committee for Standardization, 2006). The tests were performed using a Zwick/Roell Z250 mechanical testing machine with a load capacity of 200 kN and rigidity of $8 \times 10^{-3}$ mm/kN at a room temperature of 21 °C and relative humidity of 50%. In the 3 PB tests, the load was applied in the middle of the specimens with a span length of 100 mm. The tests were conducted on two sets of specimens: non-aged specimens at 28 days and specimens

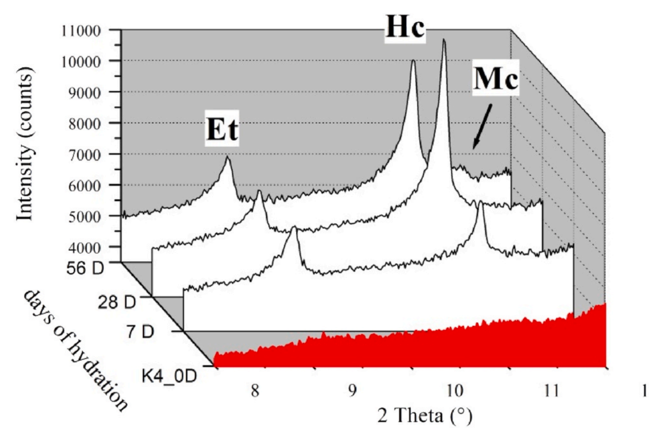


**Fig. 4.** XRD analysis illustrating the changes in the FA paste during hydration at 7, 28, and 56 days.

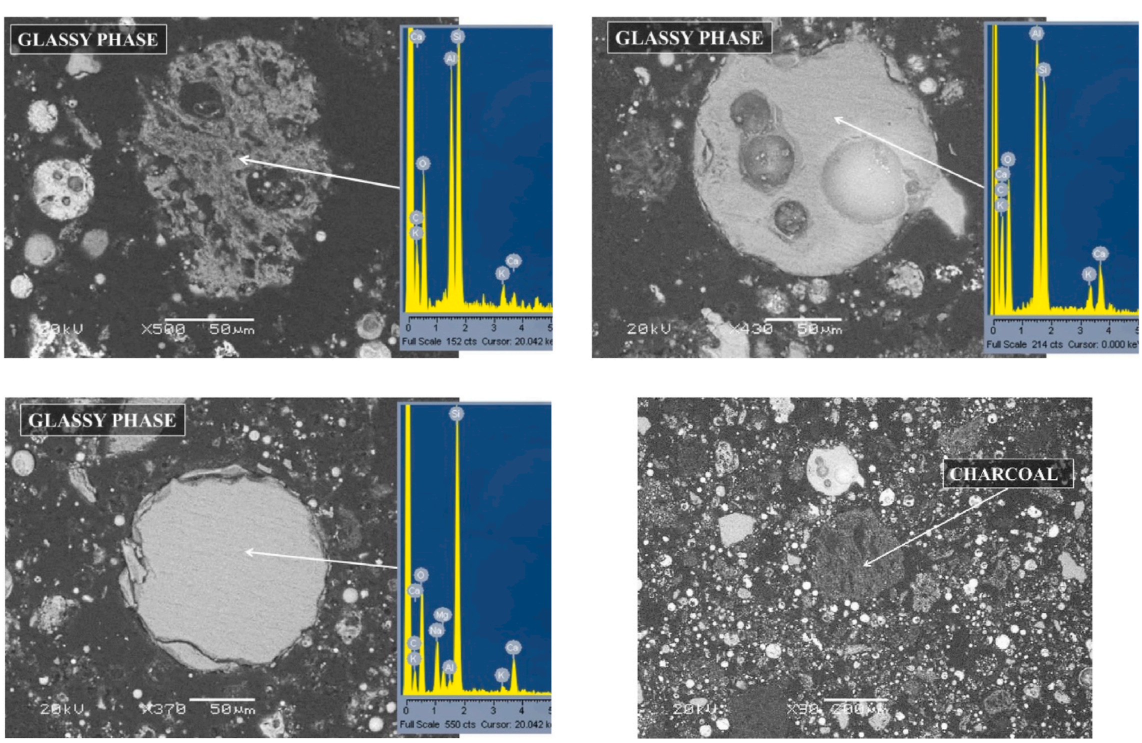


**Fig. 3.** SEM micrographs of the FA along with the results of the EDS analysis.

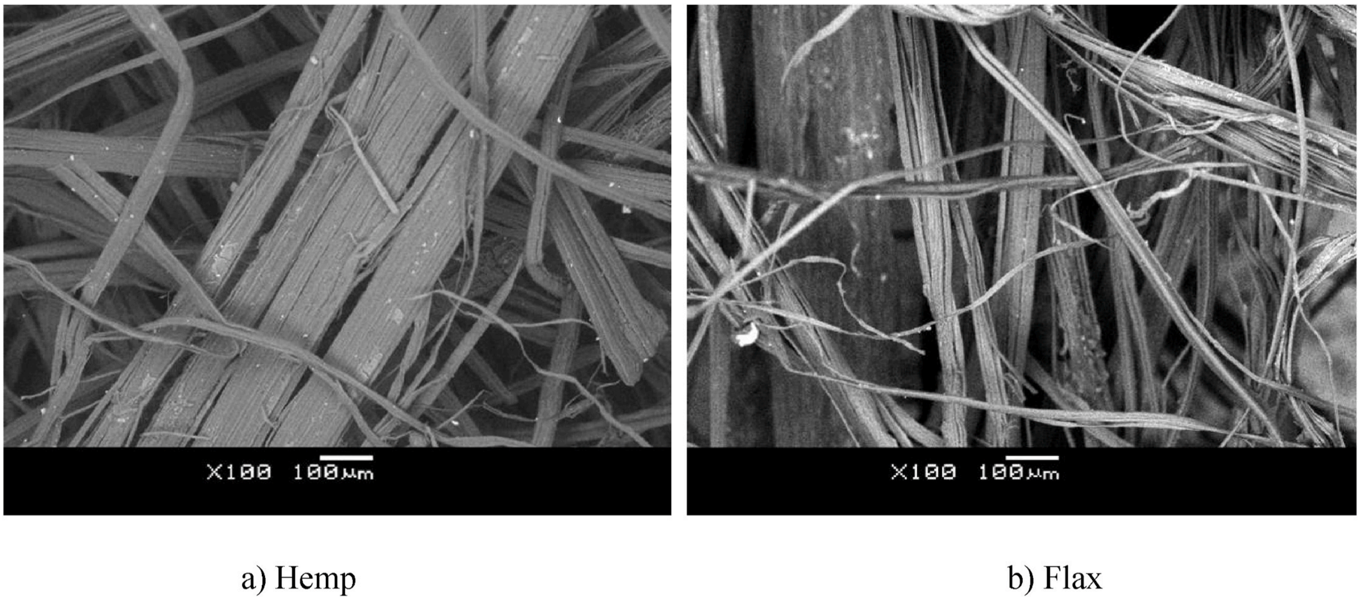


**Fig. 5.** SEM images displaying the morphology of fibers.

**Table 3**
Mortar mix designs and corresponding calculated theoretical density.

| Mixture notation | Aggregate [g] | Cement [g] | Fly ash [g] | Water [g] | Hemp fibers [g] | Flax fibers [g] | PP fibers [g] | Theoretical density [g/cm$^3$] |
|---|---|---|---|---|---|---|---|---|
| M0 | 1619 | 1619 | 0 | 648 | 0 | 0 | 0 | 2112 |
| M25 | 1619 | 1214 | 405 | 648 | 0 | 0 | 0 | 2098 |
| M50 | 1619 | 810 | 810 | 648 | 0 | 0 | 0 | 2085 |
| HM0 | 1619 | 1619 | 0 | 648 | 25 | 0 | 0 | 2107 |
| HM25 | 1619 | 1214 | 405 | 648 | 25 | 0 | 0 | 2093 |
| HM50 | 1619 | 810 | 810 | 648 | 25 | 0 | 0 | 2079 |
| FM0 | 1619 | 1619 | 0 | 648 | 0 | 25 | 0 | 2107 |
| FM25 | 1619 | 1214 | 405 | 648 | 0 | 25 | 0 | 2093 |
| FM50 | 1619 | 810 | 810 | 648 | 0 | 25 | 0 | 2079 |
| PPM0 | 1619 | 1619 | 0 | 648 | 0 | 0 | 15 | 2102 |
| PPM25 | 1619 | 1214 | 405 | 648 | 0 | 0 | 15 | 2088 |
| PPM50 | 1619 | 810 | 810 | 648 | 0 | 0 | 15 | 2074 |

exposed to accelerated aging of 56 FTCs. Compressive strength tests were also performed on all samples with three identical specimens by applying a compressive force to the 40 × 40 mm$^2$ area according to standard EN 1015-11. The compressive strength was calculated using the peak force obtained from the compressive test. From the force-displacement curves obtained in the 3 PB tests, the flexural strength ($f_{max}$) and energy absorption capacity under flexure ($E_a$) of the specimens were calculated as following equations.

$$f_{max}[\mathrm{MPa}] = (3 \times F_{max} \times L)/(2 \times b \times h^2) \quad (1)$$

$$E_a\ [\mathrm{N/m}] = \text{absorbed energy}/(b \times h) \quad (2)$$

where *b* and *h* are the specimen width and thickness respectively, L is the major span, and $F_{max}$ is the maximal force (peak of the force-displacement curve). Since after FTC aging the cross-section of the specimen was severely damaged, the corresponding area of the cross-section (b × h) was calculated using the AutoCAD® 2016 software - a photo of each specimen was taken from the front side and a picture was inserted in the software which is used to calculate the area of the specimens' cross-section. The absorbed energy in flexure was calculated as the area under the force-displacement curve up to the mid-span deflection of 5 mm of the curve.

The accelerated aging of FTCs was carried out on the specimens immersed in a 3% NaCl solution according to modified CEN/TS 12390-9 (BSI, 2016). Each specimen was placed upright in a polyethylene bottle and subjected to freezing and thawing cycles. The freezing phase lasted 16–18 h at −20 ± 2 °C, followed by a thawing phase lasting 6–8 h at 20 ± 2 °C. Visual inspections for cracks or changes were conducted after specific numbers of FTCs. Cumulative scaling (mass loss), which measures the scaled and loosened material on the specimens' surface, was collected and weighed at different stages of the FTCs. The relative scaling was calculated based on the mass of the spalled material and the mass of the specimens.

Dry bulk density following EN 1015-10 (1015-10 E. EN and 1015-10, 2007) was performed on all samples with three specimens before FTC aging, and the results were calculated based on mass and volume measurements. Liquid absorption was determined by comparing the weight of the specimens that were immersed in 3 wt% NaCl solution to their weight at ambient temperature and 50% relative humidity based on the previous studies (Wang et al., 2016, 2020). The liquid absorption (W) of the specimens was calculated as follows:

$$W\ [\%] = ((W_w - W_a)/W_a) \times 100 \quad (3)$$

$W_w$ is the weight of specimens when saturated with 3 wt% NaCl solution; $W_a$ is the weight of the specimens at ambient temperature and relative humidity of 50%.

Pore size distribution was analyzed using Mercury Intrusion Porosimetry (MIP) within a pressure range of up to 414 MPa, using an AutoPore IV 9500 porosimeter. The analysis was performed on a representative part of each sample, crushed to gradation 4–8 mm. The mortar samples were soaked in isopropanol and oven-dried (at 60 °C till constant mass) before MIP analysis. In addition, morphology, microstructure, and semiquantitative chemical analysis were performed using a JEOL 5500 LV SEM equipped with EDS. The SEM analyses were conducted in low-vacuum mode on the polished cross-sections of the mortar samples.

## 3. Results and discussion

### 3.1. Dry density values for mortars before exposure to accelerated FTS cycles

The density results are presented in Table 4, demonstrating that the measured densities of the mortar specimens are consistently 0.5–9% lower than the corresponding theoretical values (Table 3). This disparity is mostly attributed to the entrainment of air during the mixing process.

The use of FA as a cement replacement didn't show a significant impact on mortar density, resulting in a decrease of 4% at 25 wt% and 9% at 50 wt%. This was assigned to FA possessing a slightly lower density (2.600 $Mg/m^3$) compared to cement (2.800 $Mg/m^3$). The addition of fiber reinforcement to the mortars does not significantly alter the density of the specimens too, as shown in Table 4. However, it is important to note that the presence of fibers in the mortar introduces entrapped air buffers, and increases water retention, which ultimately leads to a bit further reduction in the measured density of the mortars (dos Santos Alberton et al., 2023).

The influence of FA addition is more prominent than the effect of fiber utilization, as evidenced by the similar density values measured on all FA mixes, regardless of the fiber type. In the case of mortars with FA, the hemp and flax mortars demonstrate lower densities compared to the PP mortars. This difference can be attributed to the higher water absorption and apparent porosity observed in natural fibers as compared to synthetic ones (Abdalla et al., 2023). In Tables 4 and it is observed that the addition of 25% FA has a more significant impact on the measured density in the case of natural fibers than the addition of 50% FA. The difference in densities between M25 and both HM25 and FM25 is more pronounced than between M50 and both HM50 and FM50.

### 3.2. Liquid absorption of mortar samples before and after exposure to accelerated FTS cycles

The liquid absorption properties of cementitious materials significantly impact their resistance to freeze-thaw action, as they provide insights into the volume of porous regions where water can ingress. Fig. 6 demonstrates the effects of FA and different fibers on the liquid absorption behavior of cement mortars, both before and after exposure to FTCs. Results reveal that the incorporation of FA generally increases liquid absorption, as well as the addition of fibers. It might be due to maintaining a constant water amount while substituting cement with FA leads to an excess of water due to FA's lower water reactivity compared to cement. This excess water results in increased porosity. Consequently, the outcome of replacing cement with FA is attributed to an overall increase in liquid absorption. Amritha Raj et al. (2021) also demonstrated that replacing cement with FA and adding rice straw fiber increases the rate of water absorption compared to the control mix. Furthermore, FTC exposure induces micro-cracking, leading to increased liquid absorption in all mixtures. In their study on fiber-reinforced FA concrete, Karahan and Atiş (2011) found consistent results. They observed that increasing the FA and fiber contents in all concrete mixtures led to higher values of water absorption, and sorptivity coefficient. Moreover, they noted that the addition of FA had a more significant impact on the sorptivity coefficient compared to the inclusion of PP fibers in the concrete.

**Table 4**
Comparison of mortar specimen densities in $kg/m^3$ after a 28-day curing.

| Mixtures/FA replacement level | 0% FA | 25% FA | 50% FA |
|---|---|---|---|
| M | 2104 | 2027 | 1926 |
| HM | 2116 | 1946 | 1942 |
| FM | 2081 | 1967 | 1905 |
| PPM | 2072 | 2017 | 1997 |

Notably, the addition of FA to the mixtures resulted in significantly increased liquid absorption after both pre- and post-FTS cycles. A study by Kawamura et al. (Bui et al., 2018) found that the cement hydration and pozzolanic reactions of FA proceed gradually, suggesting that longer curing periods may be necessary for complete reactions to occur. Similarly, Bayraktar et al. (Yavuz Bayraktar et al., 2021a) claimed that the use of FA in mixtures increases water absorption and porosity when they studied the effect of FA on the mechanical and durability properties of fiber-reinforced concrete. According to Fig. 6 an increase in FA content as a cement substitute led to higher liquid absorption values before FTC exposure, with liquid absorption nearly tripling in HM and FM mixtures when FA content reached 25%. Consequently, a higher volume of FA tends to increase the permeability of the mortar. Nevertheless, this trend is less pronounced in PPM with 50% FA substitution.

While the inclusion of fibers just slightly increased liquid absorption in some mixtures, their overall impact on liquid absorption was found to be negligible. Before freeze-thaw exposure, the slight increase in liquid absorption observed for HM and FM mixtures can be attributed to the porous nature of natural fibers, which may be related to the internal structure of the natural fiber, that provides channels for water penetration (Zhao et al., 2019). Santos et al. (da Costa Santos and Archbold, 2022) reported that polypropylene fibers exhibit lower permeability compared to natural fibers. Similarly, Page et al. (2017) reported that incorporating flax fibers in concrete led to increased porosity. Mortars without fiber displayed liquid absorption values of 0.8%, 1.9%, and 3.7% for M0, M25, and M50, respectively. The incorporation of hemp, flax, or PP fibers in mixtures with 50% FA reduced liquid absorption to 3.5%, 3%, and 2%, respectively, compared to the initial value of 3.7% for the corresponding fibreless mortar.

Upon exposure to FTC, the liquid absorption values for all mixtures significantly increased as shown in Fig. 6. This can be explained by crack formation in the structure of hardened cement mortars during FTC exposure, facilitating water penetration (Zhang et al., 2017b). Interestingly, the impact of FA and fiber on liquid absorption remained consistent with the pre-exposure results. Additionally, it is important to note that while the liquid absorption value for M0, HM0, FM0, and PPM0 mixtures was approximately 10%, it increased to around 15% with the addition of FA.

### 3.3. MIP test of mortar specimens before and after exposure to accelerated FTS cycles

As illustrated in Fig. 7, the incorporation of FA content in cement mortars results in a notable increase in intrusion volume, indicating a rise in the overall porosity. Specifically, the total porosity of samples without FA is approximately 15%, while it escalates to 20% and 25% for samples incorporating 25% and 50% FA, respectively (Fig. 8). Fig. 7 displays the MIP test results for the reference samples (M0, M25, and M50), representing mortars without fibers, highlighting the overall porosities and the quantities of small (<0.01 μm), medium (0.01–0.05 μm), and large (0.05–10 μm) capillaries, along with air voids (>10 μm), after 28 days of hydration.

The results indicate that the incorporation of FA leads to increased porosity in the blended mortars compared to those with only cement. The porosity rises with higher FA replacement levels. The amount of water in all mortar mixtures was constant, and cement was replaced with FA. Since FA reacts with less water than cement, this results in an excess of water, thereby increasing porosity. Also, it can be attributed to the relative size of FA, the amount of FA used as a cement substitute (Sinsiri et al., 2010), and finally, the phase composition of FA leading to delayed (latent) hydraulic reactions (Öztürk and Kılınçkale, 2023).

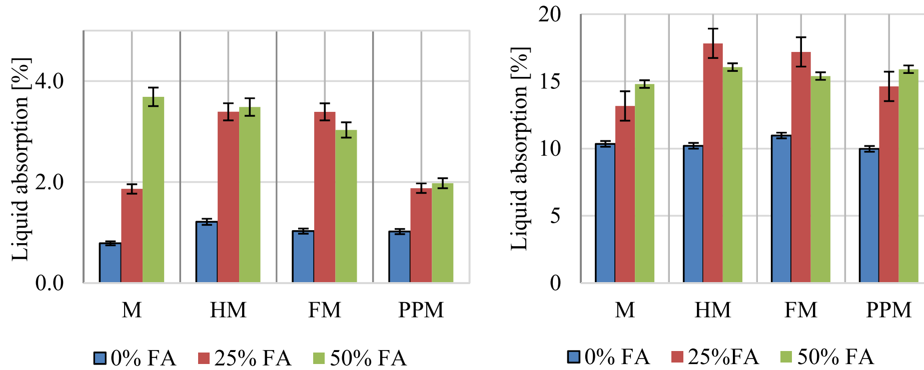


**Fig. 6.** Liquid absorption of mortar specimens: left) Pre-FTS cycles, right) Post-FTS cycles.

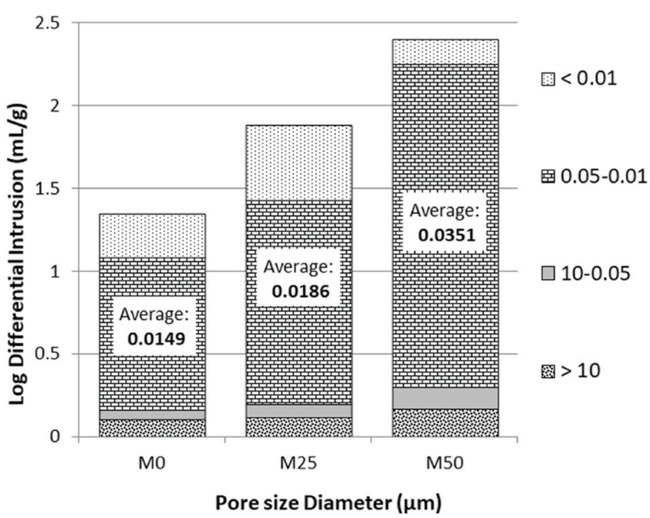


**Fig. 7.** The pore size distribution of the reference mortar mixtures measured by MIP.

Moreover, a similar trend is observed for all pore size ranges except for small pores (gel porosity). As depicted in Fig. 7, the inclusion of 50% FA replacement results in smaller amounts of small gel porosity compared to M0 and M25. The presence of 25% FA is more effective in increasing the gel porosity even compared to 0% FA, indicating better dispersion in the cement matrix, efficient de-flocculation of cement, and favorable provision of new nucleation sites for hydration products. Additionally, the average pore size diameter shows that the M25 mix has a smaller average pore diameter than M50, further confirming that 25% FA addition is more effective in reducing the average pore size due to improved dispersing and packing of FA in the M25 mortar mix.

Regarding the impact of different fibers (hemp, flax, and PP) in the mortar mixtures, no consistent trend is observed in porosity changes (Fig. 8). The influence of fiber reinforcement on the pore structure appears to depend on specific mix proportions and other material characteristics. Although minor changes, after exposure to FTCs specimens have not shown any substantial differences in porosity.

### 3.4. *Strength properties and energy absorption capacity of mortar specimens before and after exposure to accelerated FTS cycles*

#### 3.4.1. *Compressive strength*

The compressive strength of the plain mortar (M0) was 49.6 MPa. Upon the addition of hemp, flax, and PP fibers, the compressive strength of the mortar decreased to 41.3, 31.7, and 38.1, respectively (Fig. 9). The most significant decrease was observed in FM0 (36%), while HM0 and PPM0 exhibited a comparatively lower compressive strength loss (16% and 18%, respectively). This reduction in strength can be attributed to the increased air content and porosity within the matrix due to the presence of fibers (Benaniba et al., 2020), and the high water absorption of flax fibers (Moudood et al., 2019). When 25 wt% of cement was partially replaced with FA in the matrix, the compressive strength of all mixtures decreased. In the case of plain (M25) and hemp mortars (HM25), a notable decrease (up to 45% and 52%, respectively) occurred, while flax- (FM25) and PP fiber-reinforced mortars (PPM25) showed a more moderate decrease (up to 26% and 18%, respectively). Further increasing the FA content to 50 wt% only resulted in a slight reduction in compression strength for plain (M50) and PP fiber mortars (PPM50). These substantial reductions in compressive strength might be connected to the low cement content and the unfavorable influence of the

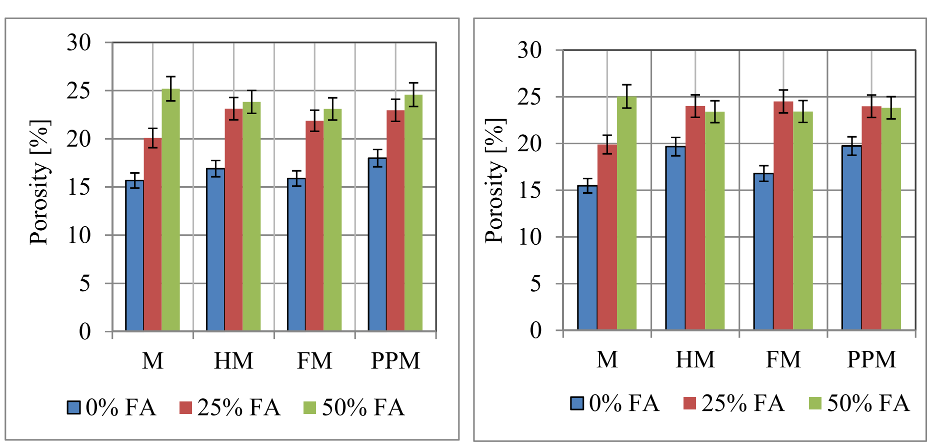


**Fig. 8.** Total porosity of mortar specimens: left) Pre-FTS cycles right) Post-FTS cycles.

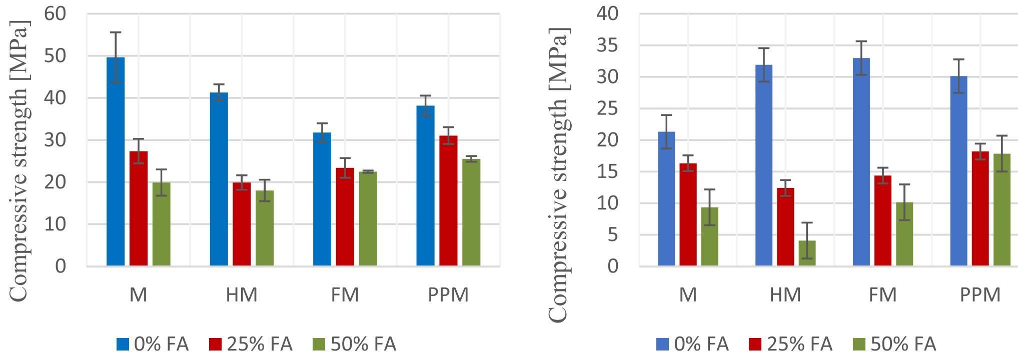


**Fig. 9.** Compressive strength of mortar specimens: left) Pre-FTS cycles, right) Post-FTS cycles.

high quantity of FA used (particle size, incomplete pozzolanic reaction) (Seifan et al., 2020; Choi et al., 2012). This aligns with the findings of Chandramohan et al. (2022), who report that compressive strength decreases as the content of FA and coconut fiber increases. Barbuta et al. (2017) also observed that increasing the quantity of cement replaced by fly ash results in a decrease in compressive strength, even with the addition of fibers. Siddique (2004) reported that replacing cement with 35%, 45%, and 55% FA reduced splitting tensile strength by 23%, 36%, and 49%, respectively. However, the addition of san fibers increased tensile strength as fiber content rose from 0.25% to 0.75%, showing its beneficial impact on FA concrete.

During accelerated FTC, the compressive strength of plain mortar (M0) significantly decreased (up to 57%), while the addition of hemp fiber to the matrix substantially reduced its strength degradation (approximately 23% strength loss). Fibers in concrete can reduce freeze-thaw damage by inhibiting crack formation, thus preserving the matrix from degradation (Zeng et al., 2023). Bayraktar et al. (Yavuz Bayraktar et al., 2021b) studied the impact of recycled concrete aggregate and FA on the mechanical and durability properties of fiber-reinforced concrete subjected to FTCs. They found that, after 50 cycles, the mixture containing 25% of recycled aggregate and 3% of PP fiber exhibited the lowest loss in compressive strength. Notably, flax fiber mortars (FM0) exhibited no compression strength loss under FTC aging.

The partial replacement of cement with FA in natural fiber mortars did not reduce the compression strength loss under FTC aging, whereas matrices with lower FA content exhibited less severe strength degradation. At 50 wt% FA content, a drastic decrease in compression strength was observed, with flax fiber mortars (FM50) showing only 30% residual strength, and hemp mortars severely disintegrating under FTCs and completely losing their compressive strength. Generally, in natural fiber specimens, the substitution of cement with FA even promoted the deterioration and degradation of the matrix, leading to a loss in compression strength. However, in plain mortars (M50) and PP fiber mortars (PPM50), the substitution of cement with FA had practically no negative effect on compression strength under FTC aging.

### 3.4.2. *Flexural strength*

Before undergoing FTCs, replacing cement with FA and adding natural fiber reinforcement to the mortar matrix resulted in a slight decrease in flexural strength, as shown in Fig. 10. However, incorporating polypropylene (PP) fibers significantly improved the flexural strength of PPM0, PPM25, and PPM50 by 21%, 9%, and 15%, respectively. These findings align with those of Siddique, who reported that replacing cement with varying percentages of FA reduced flexural strength by up to 56% (Siddique, 2004).

When subjected to accelerated aging through FTCs, all matrices exhibited general embrittlement with a decline in flexural strength (Fig. 10). However, the inclusion of fiber reinforcement significantly mitigated the rate of strength degradation. For instance, plain mortars (M0) experienced a 33% loss in flexural strength, while hemp, flax, and PP fiber mortars (HM0, FM0, PPM0) showed only 13%, 23%, and 10% losses, respectively. According to Zhang's study (Zhang et al., 2023a) fibers played a crucial role in minimizing surface denudation and the expansion of internal micro-cracks, thereby improving the frost resistance of concrete.

Partial substitution of cement with FA in plain mortar effectively curbed the degradation of flexural strength during FTCs, and in some cases, even a slight increase in flexural strength was observed for M25 and M50, which might be due to the matrix becoming more porous (Turk et al., 2022). In natural fiber-reinforced mortars, a 25 wt% replacement of cement with FA generally did not result in flexural strength degradation, whereas a higher FA content (50 wt%) led to a sharp reduction. The finding aligns with Kaplan et al.'s study (Kaplan et al., 2021) which stated that the most effective proportion of FA for enhancing freeze-thaw resistance was 25%.

Therefore, under severe environmental conditions, both hemp

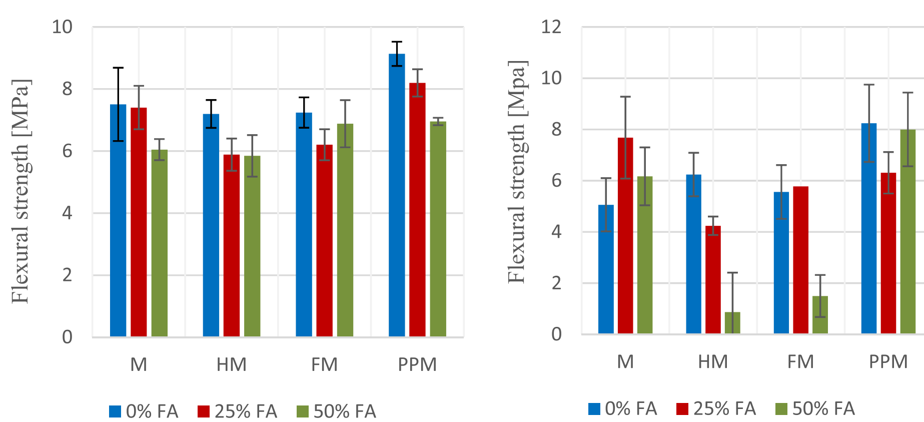


**Fig. 10.** Flexural strength of mortar specimens: left) Pre-FTS cycles right) Post-FTS cycles.

(HM25) and flax-reinforced mortars (FM25) with a 25 wt% replacement of cement with FA is a feasible alternative to traditional PP synthetic fiber mortars. Flax fiber mortars performed equally well as PP-reinforced mortars under severe environmental conditions in terms of the composite's flexural strength.

### 3.4.3. Toughness

Flexural toughness can be described as the capacity to absorb energy, in other words, it refers to the ability to endure crack opening. The incorporation of fiber into mortar mixtures enhances the post-peak plateau in stress-strain curves, resulting in a notable improvement in the energy absorption capacity, also known as toughness, of the material (Poletanovic et al., 2020). The energy absorption capacity is influenced by various fiber parameters, such as material, geometry, diameter, length, dosage, density, porosity, and water absorption, as well as the characteristics of the fiber/matrix interface, particularly the bond strength (Rostami et al., 2019). When the matrix reaches its ultimate strength capacity during flexure, it develops cracks, and the fibers actively transfer stress by bridging these cracks. This stress transfer occurs through mechanisms like fiber/matrix adhesion, friction, fiber anchorage, flexibility, and the deformation capacity of the fibers (Khan et al., 2020).

Before subjecting the mortars to FTCs, it is observed that the energy absorption capacity is significantly enhanced in the case of hemp fiber reinforcement for mortars without FA. Specifically, the energy absorption capacity of hemp fiber reinforcement increased by 348% compared to plain mortar. Similarly, flax and PP fiber reinforcement improve the energy absorption capacity by 207% and 225%, respectively, in comparison to plain mortar. The study analyzed the microstructure of hemp fiber-reinforced cement mortar samples to verify their energy absorption performance. The micrographs illustrated the good embedding of porous hemp fibers within the cement matrix and the strong contact facilitated by hydration products (Fig. 11c). These findings were consistent with previous research on hemp fibers, highlighting their triangle-like shape and cellulose composition, ensuring good adhesion to the concrete matrix. The intact hemp fibers also indicated a strong bond with the concrete matrix (Çomak et al., 2018). Ramadan et al. (2017) recommended hemp fiber as a sustainable alternative to synthetic fibers for enhancing concrete performance in harsh environments.

Incorporating fibers into concrete effectively hinders crack propagation, resulting in a notable improvement in the energy absorption capacity of concrete structures. This enhancement is particularly beneficial in applications where the reduction of shrinkage cracks is necessary (Fujikake, 2014; Alberti et al., 2014). To enhance bond formation within the cement and fiber matrix, Suhail et al. investigated the irregular surface properties of wheat straw fiber in concrete, which corresponded to a high tensile strength of 40 MPa. The bonding and bridging effects provided by wheat straw fibers within the concrete matrix improved the energy absorption capacity and toughness index, effectively inhibiting the formation of microcracks (Suhail et al., 2022). In particular, natural fibers are employed in brittle cement composites to improve and uphold their toughness (Ramakrishna and Sundararajan, 2005).

However, the situation changed when FA was partially substituted

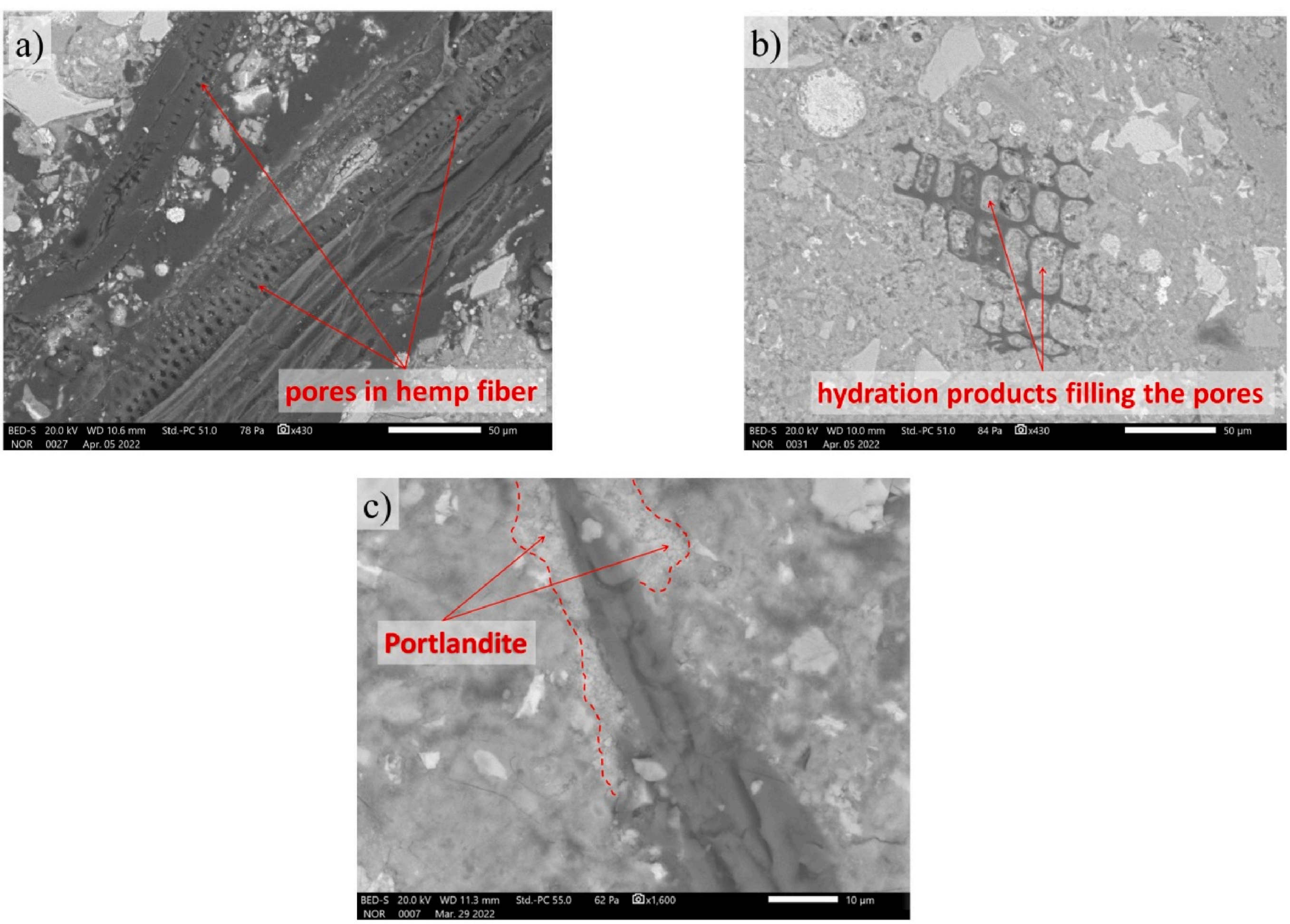


**Fig. 11.** SEM micrograph of a) porous natural hemp fiber and b) hemp fiber pores filled with hydration products in cementitious matrix c) good contact of a matrix's hydration products with fibers (fields of portlandite are labeled with dotted line).

for cement in the mixtures. The addition of FA leads to a marked decrease in the energy absorption capacity, except in the case of PPM mixtures. In the presence of PP fibers, the addition of FA enhances the energy absorption capacity of the matrix by 15% and 25% for 25 wt% and 50 wt% FA additions, respectively. Nonetheless, regardless of the FA dosage, the overall energy absorption capacity of the composite is generally adversely affected by the replacement of cement with FA. The lower energy absorption in natural fiber-reinforced concrete may be attributed to the presence of internal micro-cracks and tiny voids caused by unreacted FA (Chen et al., 2021).

The toughness of the PPM proved to be resistant to FTCs (in all cases-with or without cement substitution with FA). This could be associated with the hydrophobic characteristics of the PP fibers. It is assumed that the good contact between fibers and the matrix, as shown in Fig. 12, continued also after FTC. This notion is further supported by Poon et al. (2004), who concluded that in normal concrete, the addition of PP fibers led to a slight increase in specific toughness.

During FTC accelerated aging, there is a notable embrittlement of natural fiber matrices, causing a decrease in the post-peak plateau (Fig. 13) and a significant reduction in the energy absorption capacity under the flexural test (Fig. 13). For mortars without FA (H0 and F0), the energy absorption capacity decreased by 67% and 49%, respectively, after exposure to FTC conditions. In contrast, plain mortars (M0) only experience a 9% reduction in energy absorption, while PP fiber-reinforced mortars (PPM0) demonstrate a 9% increase in energy absorption capacity under FTCs. Natural fibers have weaker fiber-matrix adhesion in aggressive environments, resulting in lower energy absorption compared to PP fibers (Fangueiro, 2011; Bentur and Mindess, 2006).

When 25 wt% of FA is used as a partial substitution for cement in hemp- and flax-fiber mortars, there is a noteworthy improvement in energy absorption capacity during accelerated FTC aging (Fig. 14). However, with a higher FA dosage (50 wt%), no beneficial effect on energy absorption is observed, except for PP fiber-reinforced mortars.

### 3.5. *Apparent morphology and mass loss (scaling)*

The durability of mortar specimens exposed to harsh environmental conditions with FTC and deicing salts can be assessed through cumulative mass loss (surface scaling). This indicator reflects the material's resistance to degradation processes resulting from accelerated aging. Significant variations in mass loss due to scaling are evident when subjected to FTC exposure, as illustrated in Fig. 15. During the FTC, the test block undergoes freezing and swelling, causing internal cracks to expand due to excessive pressure. This accelerates the destruction of the concrete, leading to a decrease in mass. The salt freezing cycle worsens the damage, as chloride enters the cracks, increasing infiltration pressure and causing a more significant loss of mass (Zhang et al., 2023b).

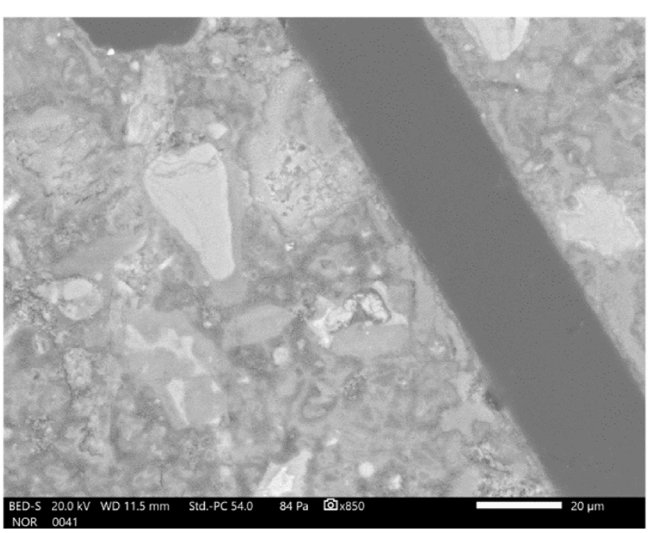


**Fig. 12.** Synthetic PP fiber in cement matrix.

Under severe environmental conditions, plain mortar (M0) experiences considerable scaling, leading to a degradation rate of 28% in mass loss after 56 FTCs. However, the partial replacement of cement with FA in plain mortars does not offer any notable benefits to their durability during aging under freeze-thaw conditions, as shown in Fig. 15. Moreover, the addition of 50% FA (M50) results in more substantial scaling compared to the addition of 25% FA in M25. This outcome can be attributed to the increasing capillary pore and lower density of mortar matrices with a higher degree of FA addition, leading to more severe degradation after freeze-thaw exposure (Ding et al., 2022). Kaplan et al. (2021) found that higher FA content leads to increased abrasion depth and mass loss. Mixes with 50% FA showed more surface spills, which contributed to higher mass losses.

In contrast, incorporating fibers into mortar mixes without FA (HM0, FM0, PPM0) has a positive effect, significantly reducing scaling under severe environmental conditions. PP fibers positively impact mortars, reducing expansion stress. This results in smaller spalling and less mass loss (Zhang et al., 2023b). The relative scaling of natural fiber mortars after 56 FTCs is only 5–8%, which falls within the same range as that of PP fiber mortars. Fiber addition acts as a bonding bridge, blocking pores and delaying crack development in concrete. This improves surface spalling and reduces mass loss during the freeze-thaw cycle (Fig. 16). According to Xiao et al. (2018), through the rapid freeze-thaw method, it was demonstrated that the addition of fibers in concrete can decrease the rate of mass loss.

The effect of adding FA to mortar mixes is most pronounced in mortars with natural fibers. Unlike synthetic fiber mortar mixes (PPM25 and PPM50), where the scaling degree is smaller compared to mortars without fibers with both 25% and 50% FA addition (M25 and M50), mortars with natural fibers exhibit different trends in the case of 25% and 50% FA addition. The substitution of 25% cement with FA in natural fiber mortars (HM25, FM25) has an unfavorable effect on surface scaling and, consequently, the composite's durability. However, when the amount of FA increases to 50%, the addition of natural fibers (HM50, FM50) has a positive effect, as surface scaling is smaller compared to both HM25 and FM25 (25% FA addition), and the 50% FA mortar without fibers (M50).

## 4. Conclusions and recommendations

This study highlights the potential of natural fiber reinforcement and partial cement replacement with FA in enhancing the durability of concrete structures in cold-weather regions. The findings demonstrate that natural fibers effectively improve FTS resistance, reduce deterioration defined by mass loss, and enhance energy absorption capacity, making them viable alternatives to synthetic fiber-reinforced mortars. Additionally, the use of FA as a cement replacement contributes to sustainable construction practices by reducing the carbon footprint. These findings offer practical guidance for designing resilient and environmentally friendly concrete structures, promoting sustainable construction methods in cold-weather environments. The results obtained in the experimental studies are summarized below.

1. Using FA as a cement replacement in mortar resulted in a 4% decrease in density at 25 wt% and a 9% decrease at 50 wt%. Additionally, FA addition significantly increased liquid absorption in pre- and post-FTS cycles, with porosity rising at higher FA replacement levels. This increase may be attributed to the gradual pozzolanic reactions of FA and the subsequent increase in the water-to-cement ratio. Incorporating FA led to decreased compressive strength, while mortars with a 25 wt% FA replacement performed well in flexural strength and toughness, especially post-FTS cycles. Higher

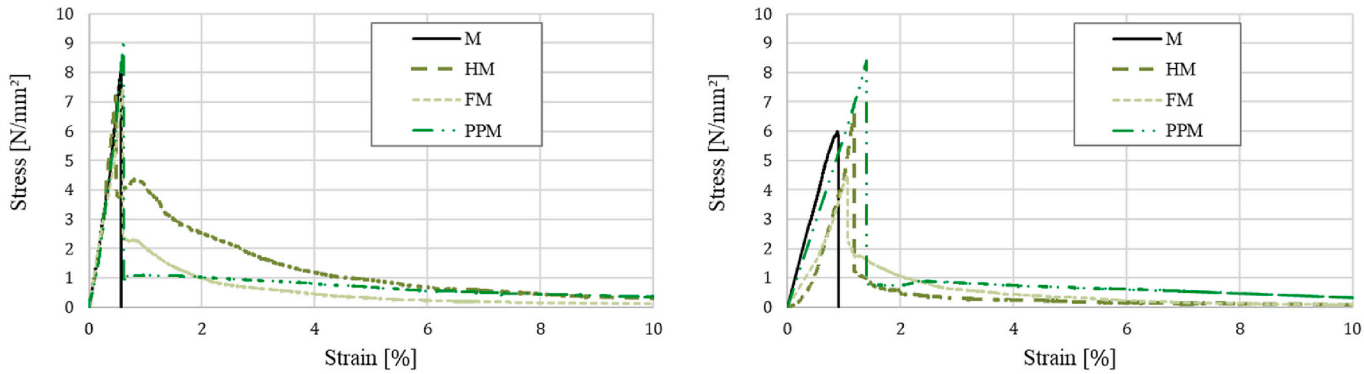


**Fig. 13.** Stress-strain curves of mortar specimens: left) Pre-FTS cycles, right) Post-FTS cycles.

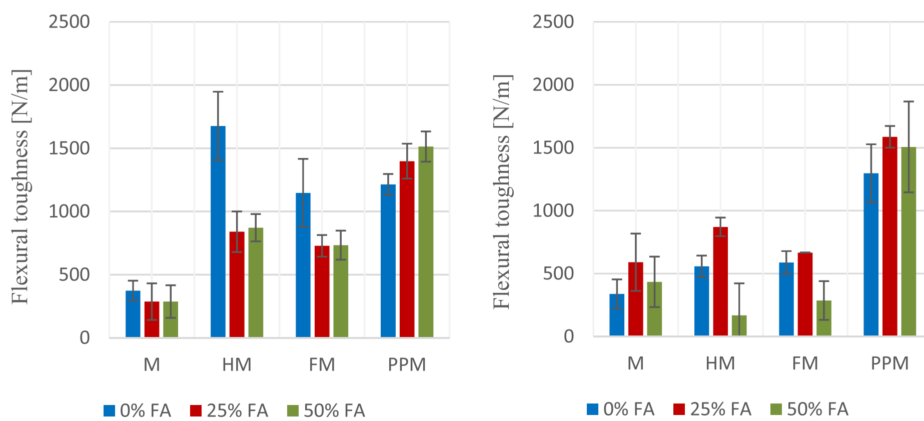


**Fig. 14.** Energy absorption capacity of mortar specimens: left) Pre-FTS cycles, right) Post-FTS cycles.

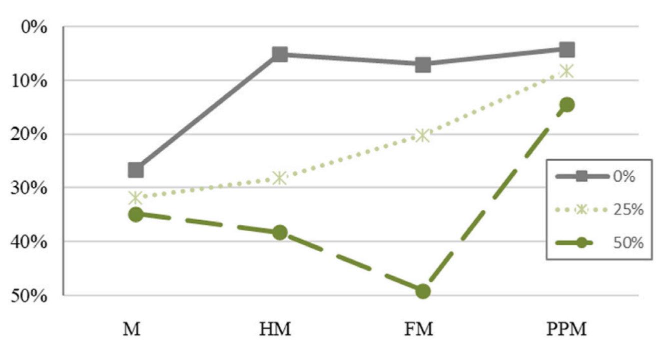


**Fig. 15.** Relative scaling of mortars under accelerated FTS Cycles.

FA content increased damage depth and mass loss, particularly at the 50% replacement level.

2. Adding fiber reinforcement to mortars does not significantly affect specimen density. Furthermore, including fibers does not show substantial differences in liquid absorption and porosity before and after exposure to FTS. However, fiber reinforcement significantly mitigates the strength degradation rate and improves toughness. Additionally, incorporating fibers into mortar mixes has a positive effect, considerably reducing scaling under FTS conditions.
3. When both FA and fibers are used, the density decreases, with the influence of FA addition being more prominent than the effect of fiber utilization. The incorporation of FA content is more significant than fibers in intrusion volume, indicating an overall rise in porosity and absorption. Under FTS conditions, both hemp and flax-reinforced mortars with a 25 wt% replacement of cement with FA is a feasible alternative to traditional PP synthetic fiber mortars. Additionally, this combination significantly mitigates degradation (mass loss) and increases energy absorption.

Although natural fibers can improve the freeze-thaw resistance, their impact on compressive strength may restrict their application in load-bearing structures. A lower dosage of fibers (0.5 vol%) could help preventing the decrease in compressive strength (Poletanovic et al., 2020). However, this in turn reduces the fibers' beneficial effect on the composite's energy absorption capacity. Thus, for an optimal compressive strength of a specific application a careful, balanced design of a compact matrix and optimal fibre dosage is inevitable (Poletanovic et al., 2020). This research focuses on applications where high energy absorption capacity, resistance to impact and dynamic loading, or crack prevention is envisaged (e.g., industrial floors, column hinge areas, tunnel linings applications, etc.). While a certain range of a compressive strength is required in these applications, it is not the primary target. Further studies are however necessary to explore how the natural fiber content or hybrid fiber combinations might enhance the frost resistance in cement-based materials. Research should especially consider pre-treatment of natural fibers (for example alkali treatment), to improve their durability under freeze-thaw cycles. Additionally, investigating the effects of combining the fibers with other admixtures (for example supplementary cementitious materials), could provide a valuable insight for improved compressive strength.

Several studies have explored the impact of longer accelerating aging on concrete durability, finding significant deterioration in mechanical properties beyond 56 cycles (Bayraktar et al., 2022; Gencel et al., 2021a; Yavuz Bayraktar et al., 2021b). Future studies should extend the number of FTCs beyond 56 to provide insights into the long-term durability of composite under severe, real harsh environmental conditions. It is also recommended to specifically investigate composites with 50% FA and

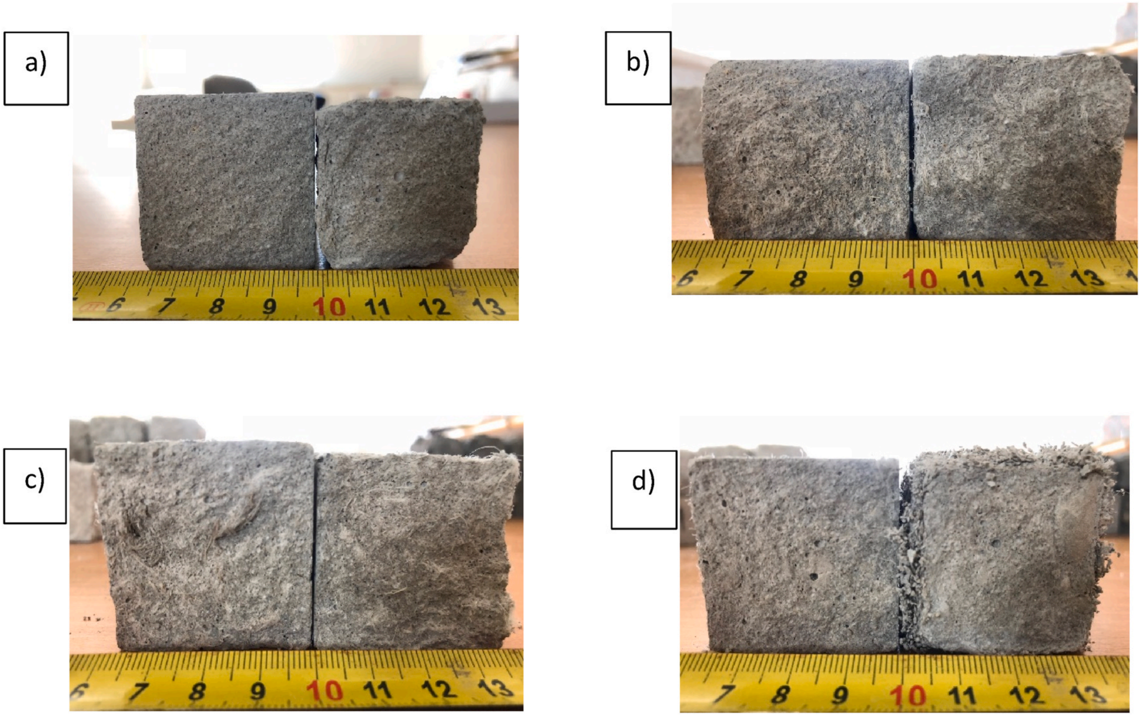


**Fig. 16.** Cross section of a) plain mortar (M0), b) hemp fiber mortar (HM0), c) flax fiber mortar (FM0), and d) PP fiber mortar (PPM0) specimens: left) Pre-FTS cycles. right) Post-FTS cycles.

above under freeze-thaw environments to obtain more insight into their resilience and performance during aging.

### CRediT authorship contribution statement

**Ildiko Merta:** Writing – original draft, Supervision, Methodology, Funding acquisition, Data curation, Conceptualization. **Vesna Zalar Serjun:** Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation. **Alenka Mauko Pranjić:** Visualization, Validation, Supervision, Resources, Methodology, Data curation. **Aljoša Šajna:** Visualization, Validation, Methodology, Formal analysis, Data curation. **Mateja Štefančič:** Validation, Methodology, Investigation, Formal analysis, Data curation. **Bojan Poletanović:** Software, Methodology, Investigation, Formal analysis. **Farshad Ameri:** Writing – original draft, Visualization, Validation. **Ana Mladenović:** Validation, Methodology, Investigation, Formal analysis, Data curation.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Acknowledgment

This research was carried out within the bilateral mobility project: *Durability of sustainable natural fiber cementitious composites in Alpine regions*, between the TU Wien (TUW) and the Slovenian National Building and Civil Engineering Institute (ZAG) funded by the Austrian Federal Ministry of Education, Science and Research (BMBWF) under the grant nr. SI 18/2016 and Slovenian Research Agency under the grant No. BI-AT/16-17-021. The authors acknowledge Slovenian Research and Innovation Agency Programme Group P2-0273 and J1-4413 project. Exclusively the mobility of the involved researchers has been funded by the programmes. The authors acknowledge TU Wien Bibliothek for financial support through its Open Access Funding Programme.

### Data availability

Data will be made available on request.

### References

1015-10 E. EN 1015-10, 2007. In: Methods of Test for Mortar for Masonry – Part 10. Determination of Dry Bulk Density of Hardened Mortar; Methods of Test for Mortar for Masonry.
Abdalla, J.A., Thomas, B.S., Hawileh, R.A., 2022. Use of hemp, kenaf and bamboo natural fiber in cement-based concrete. Mater Today Proc 65.
Abdalla, J.A., Hawileh, R.A., Bahurudeen, A., et al., 2023. A comprehensive review on the use of natural fibers in cement/geopolymer concrete: a step towards sustainability. Case Stud. Constr. Mater., e02244
Abedi, M., Hassanshahi, O., Rashiddel, A., et al., 2023. A sustainable cementitious composite reinforced with natural fibers: an experimental and numerical study. Constr Build Mater 378.
Affan, M., Ali, M., 2022. Experimental investigation on mechanical properties of jute fiber reinforced concrete under freeze-thaw conditions for pavement applications. Constr Build Mater 323.
Afroughsabet, V., Biolzi, L., Ozbakkaloglu, T., 2016. High-performance fiber-reinforced concrete: a review. J. Mater. Sci.
Al Rikabi, F.T., Sargand, S.M., Khoury, I., et al., 2018. Material properties of synthetic fiber–reinforced concrete under freeze-thaw conditions. J. Mater. Civ. Eng. 30 (6).
Alberti, M.G., Enfedaque, A., Gálvez, J.C., 2014. On the mechanical properties and fracture behavior of polyolefin fiber-reinforced self-compacting concrete. Constr Build Mater 55.
Althoey, F., Zaid, O., Majdi, A., et al., 2023. Effect of fly ash and waste glass powder as a fractional substitute on the performance of natural fibers reinforced concrete. Ain Shams Eng. J. https://doi.org/10.1016/j.asej.2023.102247.
BS-EN197-1: Cement Part 1: Composition, 2011. Specifications and conformity criteria for common cements. British Standard. November.
Baheti, V., Militky, J., 2016. Natural fibres: advances in science and technology towards industrial applications. RILEM Bookseries 12 (February).
Balaguru, P.N., Ramakrishnan, V., 1986. Freeze-thaw durablity of fiber reinforced concrete. Journal Proceedings 374–382.
Balaguru, P.N., Shah, S.P., 1992. In: Fiber-reinforced Cement Composites.
Banthia, N., Sheng, J., 1996. Fracture toughness of micro-fiber reinforced cement composites. Cem. Concr. Compos. 18 (4). SPEC. ISS.).
Barbuta, M., Bucur, R., Serbanoiu, A.A., et al., 2017. Combined effect of fly ash and fibers on properties of cement concrete. Procedia Eng.
Bayraktar, O.Y., Soylemez, H., Kaplan, G., et al., 2021. Effect of cement dosage and waste tire rubber on the mechanical, transport and abrasion characteristics of foam concretes subjected to H2SO4 and freeze–thaw. Constr Build Mater 302.

Bayraktar, O.Y., Kaplan, G., Benli, A., 2022. The effect of recycled fine aggregates treated as washed, less washed and unwashed on the mechanical and durability characteristics of concrete under MgSO4 and freeze-thaw cycles. J. Build. Eng. 48.
Benaniba, S., Driss, Z., Djendel, M., et al., 2020. Thermo-mechanical characterization of a bio-composite mortar reinforced with date palm fiber. J Eng Fiber Fabr.
Benli, A., Karataş, M., Gurses, E., 2017. Effect of sea water and MgSO4 solution on the mechanical properties and durability of self-compacting mortars with fly ash/silica fume. Constr Build Mater 146.
Bentur, A., Mindess, S., 1990. Fiber Reinforced Cementitious Composites. || Elsevier Applied Science, London and New York.
Bentur, A., Mindess, S., 2006. Fibre Reinforced Cementitious Composites. Crc Press.
Betterman, L.R., Ouyang, C., Shah, S.P., 1995. Fiber-matrix interaction in microfiber-reinforced mortar. Adv. Cement Base Mater. 2 (2).
Bogoeva-Gaceva, G., Avella, M., Malinconico, M., et al., 2007. Natural fiber eco-composites. Polym. Compos. 28 (1).
British Standards Institution, 2013. BS EN 196-2:2013: Method of Testing Cement Part 2 : Chemical Analysis of Cement. BSI Standards Limited.
Bsi, P.D., 2016. CEN/TS 12390-9: 2016; testing hardened concrete—freeze-thaw resistance with de-icing salts—scaling. CEN (European Commitee for Standardization): Brussels, Belgium 36.
Bui, P.T., Ogawa, Y., Kawai, K., 2018. Long-term pozzolanic reaction of fly ash in hardened cement-based paste internally activated by natural injection of saturated Ca(OH)2 solution. Materials and Structures/Materiaux et Constructions 51 (6).
Chandramohan, N., Loganayagan, S., Sridhar, S., et al., 2022. Investigation on strength of concrete with addition of fly ash and natural fiber. Materials Research Proceedings.
Chen, W., Shaikh, F., Li, Z., et al., 2021. Dynamic compressive properties of high volume fly ash (HVFA) concrete with nano silica. Constr Build Mater 301.
Choi, S.J., Lee, S.S., Monteiro, P.J.M., 2012. Effect of fly ash fineness on temperature rise, setting, and strength development of mortar. J. Mater. Civ. Eng. 24 (5).
Chokshi, S., Parmar, V., Gohil, P., et al., 2022. Chemical composition and mechanical properties of natural fibers. J. Nat. Fibers.
Çomak, B., Bideci, A., Salli Bideci, Ö., 2018. Effects of Hemp Fibers on Characteristics of Cement Based Mortar, vol. 169. Constr Build Mater.
da Costa Santos, A.C., Archbold, P., 2022. Suitability of surface-treated flax and hemp fibers for concrete reinforcement. Fibers 10 (11).
De Souza Rodrigues, C., Ghavami, K., Stroeven, P., 2006. Porosity and water permeability of rice husk ash-blended cement composites reinforced with bamboo pulp. J. Mater. Sci.
Ding, M., Zhang, F., Ling, X., et al., 2018. Effects of freeze-thaw cycles on mechanical properties of polypropylene Fiber and cement stabilized clay. Cold Reg. Sci. Technol. 154.
Ding, Z., Quy, N.X., Kim, J., et al., 2022. Evaluations of frost and scaling resistance of fly ash concrete in terms of changes in water absorption and pore structure under the accelerated carbonation conditions. Constr Build Mater 345.
Dong, F., Wang, H., Yu, J., et al., 2021. Effect of freeze–thaw cycling on mechanical properties of polyethylene fiber and steel fiber reinforced concrete. Constr Build Mater 295.
dos Santos Alberton, K., do Nascimento, C.B., Cavalheiro, R.B., et al., 2023. Properties of coconut fiber-reinforced mortars for sustainable solutions. Journal of Building Pathology and Rehabilitation 8 (1), 44.
European Committee for Standardization, 2006. EN 1015-11 methods of test for mortar for masonry. Part 11: Determination of Flexural and Compressive Strength of Hardened Mortar 3 (1).
Fagerlund, G., 1976. In: Studies of the Destruction Mechanism at Freezing of Porous Materials.
Fangueiro, R., 2011. Fibrous and Composite Materials for Civil Engineering Applications. Elsevier.
Filho, J.D.A.M., Silva, F.D.A., Toledo Filho, R.D., 2013. Degradation kinetics and aging mechanisms on sisal fiber cement composite systems. Cem. Concr. Compos. 40.
Fritsche, U., Brunori, G., Chiaramonti, D., et al., 2020. Future Transitions for the Bioeconomy towards Sustainable Development and a Climate-Neutral Economy—Knowledge Synthesis Final Report, vol. 10. Publications Office of the European Union, Luxembourg, 667966.
Fujikake, K., 2014. Impact performance of ultra-high performance fiber reinforced concrete beam and its analytical evaluation. Int. J. Prot. Struct.
Gencel, O., Benli, A., Bayraktar, O.Y., et al., 2021a. Effect of waste marble powder and rice husk ash on the microstructural, physico-mechanical and transport properties of foam concretes exposed to high temperatures and freeze–thaw cycles. Constr Build Mater 291.
Gencel, O., Yavuz Bayraktar, O., Kaplan, G., et al., 2021b. Characteristics of hemp fibre reinforced foam concretes with fly ash and Taguchi optimization. Constr Build Mater 294.
Jacobsen, S., Sellevold, E.J., 1994. Frost/salt scaling testing of concrete-importance of absorption during test. Nord. Concr. Res. 14 (1), 1994.
Jin, S., Yang, Y., Sun, Y., et al., 2023. Experimental research on anti-freezing and thawing performance of basalt fiber reinforced fly ash concrete in the corrosive conditions. KSCE J. Civ. Eng. 1–16.
Kaplan, G., Bayraktar, O.Y., Memis, S., 2021. Effect of high volume fly ash and micro-steel fiber on flexural toughness and durability properties in self-compacting lightweight mortar (SCLM). Constr Build Mater 307.
Karahan, O., Atiş, C.D., 2011. The durability properties of polypropylene fiber reinforced fly ash concrete. Mater. Des. 32 (2), 1044–1049.
Karbhari, V.M., 2002. Response of fiber reinforced polymer confined concrete exposed to freeze and freeze-thaw regimes. J. Compos. Construct. 6 (1).
Khan, M., Rehman, A., Ali, M., 2020. Efficiency of silica-fume content in plain and natural fiber reinforced concrete for concrete road. Constr Build Mater 244.
Knutsson, A., Luping, T., Lindvall, A., 2011. Freeze/thaw durability of concrete with fly ash. Nord. Concr. Res. 43 (1).
Komuraiah, A., Kumar, N.S., Prasad, B.D., 2014. Chemical composition of natural fibers and its influence on their mechanical properties. Mech. Compos. Mater. 50 (3).
Kothari, A., Habermehl-Cwirzen, K., Hedlund, H., et al., 2020. A review of the mechanical properties and durability of ecological concretes in a cold climate in comparison to standard ordinary portland cement-based concrete. Materials.
Liu, Z., Kang, Y., Hansen, W., 2013. Cryogenic suction pump: a major mechanism of salt frost deterioration. Proceedings–Understanding the fundamental properties of concrete. Celebrating Prof Erik J Sellevolds 75, 25–26.
Merta, I., 2016. Hemp Fibres—A Promising Reinforcement for Cementitious Materials. RILEM Bookseries.
Merta, I., Poletanovic, B., Kopecsko, K., 2017. Durability of natural fibres within cement-based materials-review. Concrete Structures Journal of the Hungarian Group of fib (Federation International de Beton) 18, 10–16.
Mohr, B.J., Nanko, H., Kurtis, K.E., 2005. Durability of kraft pulp fiber-cement composites to wet/dry cycling. Cem. Concr. Compos. 27 (4).
Moudood, A., Rahman, A., Öchsner, A., et al., 2019. Flax fiber and its composites: an overview of water and moisture absorption impact on their performance. J. Reinforc. Plast. Compos. 38 (7).
Mu, R., Miao, C., Luo, X., et al., 2002. Interaction between loading, freeze-thaw cycles, and chloride salt attack of concrete with and without steel fiber reinforcement. Cem Concr Res 32 (7).
Onuaguluchi, O., Banthia, N., 2016. Plant-based natural fibre reinforced cement composites: a review. Cem. Concr. Compos. 68.
Öztürk, H., Kılınçkale, F.M., 2023. The effect of wetting-drying/freezing-thawing cycles on properties of non-air entrained fly ash substituted cement-based composites. Ceram. Int. 49 (7).
Page, J., Khadraoui, F., Boutouil, M., et al., 2017. Multi-physical properties of a structural concrete incorporating short flax fibers. Constr Build Mater 140.
Page, J., Khadraoui, F., Gomina, M., et al., 2021. Enhancement of the long-term mechanical performance of flax fiber-reinforced cementitious composites by using alternative binders. J. Build. Eng. 40.
Poletanovic, B., Dragas, J., Ignjatovic, I., et al., 2020. Physical and mechanical properties of hemp fibre reinforced alkali-activated fly ash and fly ash/slag mortars. Constr Build Mater 259.
Poon, C.S., Shui, Z.H., Lam, L., 2004. Compressive behavior of fiber reinforced high-performance concrete subjected to elevated temperatures. Cem Concr Res 34 (12).
Powers, T.C., 1958. In: The Physical Structure and Engineering Properties of Concrete.
Powers, T.C., Helmuth, R.A., 1953. Theory of Volume Changes in Hardened Portland-Cement Paste during Freezing. Highway research board proceedings.
Raj, A., Sathyan, D., Mini, K.M., 2021. Performance evaluation of natural fiber reinforced high volume fly ash foam concrete cladding. Advances in Concrete Construction 11 (2).
Ramachandran, K.S., Feldman, R., 1996. Concrete admixtures handbook. Properties, Science, and Technology, second ed.
Ramadan, R., Saad, G., Awwad, E., et al., 2017. Short-term durability of hemp fibers. Procedia Eng.
Ramakrishna, G., Sundararajan, T., 2005. Studies on the durability of natural fibres and the effect of corroded fibres on the strength of mortar. Cem. Concr. Compos.
Richardson, A.E., Coventry, K.A., Wilkinson, S., 2012. Freeze/thaw durability of concrete with synthetic fibre additions. Cold Reg. Sci. Technol. 83–84.
Rostami, R., Zarrebini, M., Sanginabadi, K., et al., 2019. The effect of specific surface area of macro fibers on energy absorption capacity of concrete. J. Textil. Inst. 110 (5).
Seifan, M., Mendoza, S., Berenjian, A., 2020. Mechanical properties and durability performance of fly ash based mortar containing nano- and micro-silica additives. Constr Build Mater 252.
Setzer, M.J., 2001. Micro-ice-lens formation in porous solid. J. Colloid Interface Sci. 243 (1).
Siddique, R., 2004. Properties of concrete incorporating high volumes of class F fly ash and san fibers. Cem Concr Res 34 (1).
Sinsiri, T., Chindaprasirt, P., Jaturapitakkul, C., 2010. Influence of fly ash fineness and shape on the porosity and permeability of blended cement pastes. Int. J. Miner. Metall. Mater. 17 (6).
Song, H., Liu, J., He, K., et al., 2021. A comprehensive overview of jute fiber reinforced cementitious composites. Case Stud. Constr. Mater. 15.
Suhail, M.A., Shrivastava, S., Paritosh, K., et al., 2022. Advances in applications of cereal crop residues in green concrete technology for environmental sustainability: a review. Agriculture (Switzerland).
Tang, S.W., Yao, Y., Andrade, C., et al., 2015. Recent durability studies on concrete structure. Cem Concr Res.
Tian, H., Zhang, Y.X., Yang, C., et al., 2016. Recent advances in experimental studies of the mechanical behaviour of natural fibre-reinforced cementitious composites. Struct. Concr. 17 (4).
Tolêdo Filho, R.D., Scrivener, K., England, G.L., et al., 2000. Durability of alkali-sensitive sisal and coconut fibres in cement mortar composites. Cem. Concr. Compos. 22 (2).
Tolêdo Romildo, D.F.D., Ghavami, K., England, G.L., et al., 2003. Development of vegetable fibre-mortar composites of improved durability. Cem. Concr. Compos. 25 (2).
Tonoli, G.H.D., Joaquim, A.P., Arsne, M.A., et al., 2007. Performance and durability of cement based composites reinforced with refined sisal pulp. Mater. Manuf. Process. 22 (2).
Turk, K., Kina, C., Nehdi, M.L., 2022. Durability of engineered cementitious composites incorporating high-volume fly ash and limestone powder. Sustainability 14 (16).
Valenza, J.J., Scherer, G.W., 2006. Mechanism for salt scaling. J. Am. Ceram. Soc. 89 (4).

Verbeck, G.J., Klieger, P., 1957. Studies of'salt'scaling of concrete. Highway Research Board Bulletin 150.
Wang, Y., Li, V.C., Backer, S., 1990. Tensile properties of synthetic fiber reinforced mortar. Cem. Concr. Compos. 12 (1).
Wang, Y., Gong, F., Zhang, D., et al., 2016. Mesoscale study of water transport in mortar influenced by sodium chloride. Journal of Asian Concrete Federation 2 (1).
Wang, Y., Ueda, T., Gong, F., et al., 2020. Experimental examination of electrical characteristics for portland cement mortar frost damage evaluation. Materials 13 (5).
Wang, R., Hu, Z., Li, Y., et al., 2022. Review on the Deterioration and Approaches to Enhance the Durability of Concrete in the Freeze–Thaw Environment. Constr Build Mater.
Xiao, Q., Hao, S., Ning, X.L., 2018. Experimental study on the frost resistance of hybrid fiber concrete. Concrete 2018, 54–57.
Yan, L., Kasal, B., Huang, L., 2016. A review of recent research on the use of cellulosic fibres, their fibre fabric reinforced cementitious, geo-polymer and polymer composites in civil engineering. Compos. B Eng. 92.
Yavuz Bayraktar, O., Salem Taher Eshtewı, S., Benli, A., et al., 2021a. The impact of RCA and fly ash on the mechanical and durability properties of polypropylene fibre-reinforced concrete exposed to freeze-thaw cycles and MgSO4 with ANN modeling. Constr Build Mater 313.
Yavuz Bayraktar, O., Salem Taher Eshtewı, S., Benli, A., et al., 2021b. The impact of RCA and fly ash on the mechanical and durability properties of polypropylene fibre-reinforced concrete exposed to freeze-thaw cycles and MgSO4 with ANN modeling. Constr Build Mater 313.
Zakaria, M., Ahmed, M., Hoque, M.M., et al., 2015. Effect of jute yarn on the mechanical behavior of concrete composites. SpringerPlus 4, 1–8.
Zeng, Q., Li, K., Fen-chong, T., et al., 2010. A study of the behaviors of cement-based materials subject to freezing. 2010 International Conference on Mechanic Automation and Control Engineering. IEEE, pp. 1611–1616.
Zeng, Y., Meng, S., Xu, H., et al., 2023. Strength attributes of fiber-reinforced lightweight aggregate concrete incorporating Lytag ceramsite under freeze-thaw environment. J. Build. Eng. 65.
Zhang, P., Wittmann, F.H., Vogel, M., et al., 2017a. Influence of freeze-thaw cycles on capillary absorption and chloride penetration into concrete. Cem Concr Res 100.
Zhang, P., Wittmann, F.H., Vogel, M., et al., 2017b. Influence of freeze-thaw cycles on capillary absorption and chloride penetration into concrete. Cem Concr Res 100.
Zhang, D., Mao, M., Zhang, S., et al., 2019. Influence of stress damage and high temperature on the freeze–thaw resistance of concrete with fly ash as fine aggregate. Constr Build Mater 229.
Zhang, Z., Ji, Y., Ji, W., 2023a. Durability performance investigation for engineering fiber cementitious composites (ECC): review. Polymers.
Zhang, J., Wu, Y., Ren, B., 2023b. Experimental study on frost resistance of hybrid fiber fly ash concrete. AIP Adv. 13 (6).
Zhao, K., Xue, S., Zhang, P., et al., 2019. Application of natural plant fibers in cement-based composites and the influence on mechanical properties and mass transport. Materials 12 (21).
Zhao, C., Wang, Z., Zhu, Z., et al., 2023. Research on Different Types of Fiber Reinforced Concrete in Recent Years: an Overview. Constr Build Mater.
Zheng, X., Wang, Y., Zhang, S., et al., 2022. Research progress of the thermophysical and mechanical properties of concrete subjected to freeze-thaw cycles. Constr Build Mater.
Ziane, S., Khelifa, M.-R., Mezhoud, S., 2020. A study of the durability of concrete reinforced with hemp fibers exposed to external sulfatic attack. Civ. Environ. Eng. Rep. 30 (2).