



Durability of cement mortars containing fine demolition wastes as supplementary cementitious materials

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ABSTRACT

The study addresses the durability performance of cementitious mortars containing 20 % recycled powders derived from construction and demolition waste, specifically concrete screenings, mixed screenings, and slurries from the washing of recycled aggregates. Uncalcined and thermally activated at 500°C, the powders were evaluated for their potential as supplementary cementitious materials. Key durability parameters analyzed include freeze-thaw resistance, fire resistance and sulfuric acid corrosion resistance, along with capillary absorption and drying shrinkage. Analysis of Variance (ANOVA) was used to assess the influences of various factors on the durability properties. The results demonstrate that the type of waste has a significant impact on the durability of mortars, with ceramic-containing specimens exhibiting superior performance in comparison to those comprising pure concrete powders. Capillary absorption emerged as an important factor for resistance to extreme temperatures. Furthermore, the findings of this study demonstrate that thermal activation of waste powders does not enhance the durability parameters, and that drying shrinkage shows no significant impact on durability. This study fills a knowledge gap in the durability of cementitious materials incorporating recycled powders and highlights the potential of ceramic-containing powders in sustainable cement production.

1. Introduction

In recent years, the generation of construction and demolition waste (CDW) has been steadily increasing due to population growth and urbanization. This inert waste has found successful application as recycled coarse and fine aggregates [1–5], nevertheless the presence of cement mortar adhering to recycled aggregates significantly diminishes their quality. Addressing this issue through polishing and washing operations gives rise to the generation of secondary waste materials, such as screening fines or washing mud. These materials are characterised by their fine granulometry, which renders them unsuitable for use as aggregates [6]. To mitigate this challenge, researchers are exploring the potential of recycled concrete powders (RCP) as supplementary cementitious materials (SCMs) to partially replace cement clinker and reduce carbon footprint of cement production [7–10]. Among these, the use of thermally activated concrete powders has shown significant promise, as thermal treatment partially restores the hydraulic properties of the powders through the decomposition of portlandite and C-S-H gel [11–14].

Despite numerous studies on the use of CDW as SCMs in eco-cement

production, the majority of these studies have focused on the fresh and mechanical properties of the resulting binders. However, there is currently a lack of research addressing their durability characteristics, which are critical for practical applications. The effective utilization of recycled concrete powders requires a comprehensive understanding of their ability to withstand adverse environmental conditions.

Most publications on the durability of cementitious materials incorporating rehydrated RCP focus on resistance to chloride penetration and, to a lesser extent, carbonation. Researchers from the University of Lisbon investigated chloride migration and carbonation in concrete specimens containing RCP activated at 650°C in amounts ranging from 5 % to 100 % of Portland cement. They concluded that the durability of concrete with rehydrated RCP is comparable to conventional Portland cement concrete due to its refined pore structure [15,16]. Similar findings were reported by Cantero et al. [17], who observed enhanced chloride and carbonation resistance in concrete specimens with 10 % uncalcined RCP. Hu et al. [18] demonstrated that mortars with 50 % RCP calcined at 800°C achieved resistance to chloride penetration comparable to reference specimens. However, contrasting results were found in the study by Xu et al. [19], where concrete made with 100 %

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RCP activated at 650°C exhibited reduced durability due to increased porosity. Sun et al. [20] observed that the incorporation of 30 % and 50 % uncalcined waste powders into cement mortar specimens resulted in increased chloride penetration due to matrix loosening caused by inert particles. Similar results were reported by Ma et al. [21] for concrete specimens. However, heat treatment of the recycled powders improved chloride penetration resistance, although it remained lower than that of the reference sample. Wu et al. [22] investigated concrete specimens containing a blend of uncalcined recycled concrete powder and recycled ceramic brick powder. Their findings indicated that recycled powders positively affect chloride penetration resistance, while their positive impact on carbonation resistance persists only when the cement substitution rate is limited to 15 %. Furthermore, Gao et al. [23] reported that the degree of carbonation in cement mortars increases as the substitution level of Portland cement by uncalcined RCP rises.

Meanwhile, critical durability parameters such as freeze-thaw resistance, fire resistance, and sulfuric acid corrosion resistance of cementitious materials containing RCP remain largely underexplored.

Algourdin et al. investigated the resistance of mortar specimens containing recycled powders activated at 500°C to high-temperature exposure [24] and freeze-thaw cycles [25]. Their findings revealed that after exposure to 200°C, the mechanical properties of the mortar samples remained virtually unchanged. However, a significant loss of strength occurred upon exposure to 500°C, with the strength loss decreasing in proportion to the degree of Portland cement substitution by thermally activated recycled powders increased. Additionally, following 100 freeze-thaw cycles at temperatures ranging from −20°C to +20°C, all specimens exhibited an increase in compressive strength due to ongoing hydration, and the mass loss of recycled cement specimens was comparable to that of the reference specimen.

Rocha et al. [7] conducted a comprehensive literature review on the use of RCP as SCMs, identifying only three studies that investigated freeze-thaw resistance [23,26,27]. All of these studies focused on uncalcined recycled powders and reported a decline in freeze-thaw resistance of materials containing RCPs, attributed to microstructure loosening.

Belkadi et al. [28] investigated cement mortars with 10 %, 15 %, and 20 % uncalcined RCP and observed a decline in sulfuric acid corrosion resistance with increasing cement replacement levels attributed to the porous nature of the recycled powder, irregular morphology, and poor adhesion.

Thus, it is evident that the durability parameters of cement containing thermally activated RCPs, such as resistance to freeze-thaw during seasonal changes in the temperate zone, fire resistance in case of fire and resistance to corrosion by sulfuric acid formed in wastewater as a result of the vital activity of microorganisms and in drops of acid rain, have not yet been studied.

Although capillary absorption and drying shrinkage are not durability properties in themselves, they are important indicators that can influence durability properties indirectly. High capillary absorption usually indicates higher porosity and permeability, which can make a material more vulnerable to durability issues like chemical attack, freeze-thaw damage, or reinforcement corrosion. On the other hand, larger regular air voids can act as pressure relief inclusions, allowing expanding ice to move into these voids, which reduces internal pressure and mitigates freeze-thaw damage [29]. Excessive shrinkage also indicates higher permeability, as it can lead to the development of cracks that compromise durability by allowing the ingress of harmful agents. These two properties have been relatively well described in the context of RCP use. While the effect of increasing RCP content on capillary absorption is generally agreed upon, with studies usually showing an increase in capillary absorption [15,16,19,21,30], the results regarding the effect on drying shrinkage are contradictory. Some studies suggest that recycled powders reduce shrinkage due to lower reactivity, reduced gel pore content, and higher particle hardness [20,31], while others report an increase in drying shrinkage due to higher porosity [32,33].

This study extends the research results published earlier [34] by examining the durability performance of cementitious mortars containing 20 % recycled powders derived from screening of recycled concrete aggregates, recycled mixed aggregates and mud from recycled mixed aggregates washing, both untreated and thermally activated at 500°C. Durability parameters such as freeze-thaw resistance, fire resistance, and sulfuric acid corrosion resistance were analyzed alongside drying shrinkage and capillary absorption. The significance of this work lies in addressing the critical knowledge gap regarding the durability of cementitious materials incorporating recycled CDW powders, particularly after thermal activation, a field with limited prior research. Notably, this study is one of the first to explore the use of mixed powders containing ceramic particles, an area that has received little to no attention in existing literature.

2. Materials

In this study, three types of fine demolition waste provided by the Belgian company Tradecowall were used for the preparation of mortar specimens. Tradecowall processes inert waste into recycled coarse and fine aggregates, involving several screening stages that separate the aggregates into different fractions. Substandard fractions from this screening process, which are unsuitable as aggregates, are stored on-site as final waste. Additionally, the washing of recycled aggregates generates wash mud, which is also stored on-site.

The three types of demolition waste fines utilized were:

- Concrete Screening Waste (CS): This consisted of concrete fines mixed with minor impurities, including metal reinforcement debris.
- Mixed Screening Waste (MS): This comprised approximately 60 % concrete and 40 % ceramics, glass, and stones, with minor impurities of organic materials such as wood, bitumen, polymers, and fabrics. The ceramic debris in MS originated from unseparated demolition waste and was not compositionally controlled. However, it predominantly consisted of ceramic bricks, wall and floor tiles, with potential contributions from roof tiles and, to a lesser extent, sanitary porcelain.
- Washing Mud (WM): This was a dark gray mass resulting from the aggregates washing process.

The waste materials underwent drying at 105°C until reaching constant mass, followed by a two-stage crushing process. Preliminary crushing was done using a jaw crusher, and final crushing employed an impact mill with a 2 mm mesh bottom sieve. Post-crushing, the materials were sieved using a 125 µm mesh sieve, resulting in two fractions: 0/125 µm and 0.125/2 mm. Only the 0/125 µm fraction was used in this study. The powders dried at 105°C were labeled as CS105, MS105, and WM105 for concrete screenings, mixed screenings, and washing mud respectively.

Subsequently, these materials were calcined in a muffle furnace at 500°C for 2 hours with a heating rate of 10 °C/min and allowed to cool naturally in a closed furnace. The calcined powders were labeled as CS500, MS500 and WM500 for concrete screenings, mixed screenings, and washing mud respectively.

For the preparation of mortar specimens, the following additional materials were used: Portland cement CEM I 52.5 R provided by the French manufacturer Vicat, CEN Standard Sand conforming to EN 196–1 [35] and tap water. Table 1 provides the chemical composition and the key physical properties of the raw materials utilized in this study.

The mineralogical composition of MS and WM was predominantly quartz, whereas CS contained both quartz and calcite in equal proportions. Calcite was also present in MS and WM, albeit in smaller quantities compared to CS, with WM exhibiting the lowest calcite content. Furthermore, the significant amounts of feldspars and muscovite were identified in all the examined wastes, with WM showing the highest concentration of these minerals. In terms of mineralogical

Table 1

Chemical and physical properties of the powders used in the study.

Property	CEM I	CS	MS	WM
SiO ₂ (mass-%)	16.07	28.42	41.94	44.60
Al ₂ O ₃ (mass-%)	3.91	5.90	8.10	9.42
Fe ₂ O ₃ (mass-%)	3.58	4.12	5.51	5.83
CaO (mass-%)	66.72	33.29	21.24	17.76
MgO (mass-%)	1.45	1.63	1.11	1.37
TiO ₂ (mass-%)	0.37	0.45	0.77	0.81
MnO (mass-%)	0.08	0.12	0.11	0.11
Na ₂ O (mass-%)	0.26	0.35	0.47	0.30
K ₂ O (mass-%)	1.18	1.25	2.15	2.30
P ₂ O ₅ (mass-%)	0.39	0.17	0.44	0.34
SO ₃ (mass-%)	3.88	0.80	2.56	1.27
LOI (mass-%)*	2.10	23.50	15.60	15.90
Specific gravity before calcination (g/cm ³)	3.045	2.444	2.547	2.509
Specific gravity after calcination at 500°C (g/cm ³)	—	2.527	2.591	2.613
Blaine SSA before calcination (cm ² /g)	5418	5620	5316	5436
Blaine SSA after calcination at 500°C (cm ² /g)	—	5539	5049	5105

* Loss on ignition at 950 °C

differences, MS did not contain dolomite, in contrast to CS and WM. Similarly, gypsum was absent in CS, distinguishing it from the other two wastes. Notably, CS was the only waste that included clay minerals, specifically chlorites.

3. Experimental methods

3.1. Compressive strength

The compressive strength of the mortars containing demolition wastes were assessed using 40x40x160 mm mortar bars, prepared according to ISO 679:2009 [36]. The water-to-binder (w/b) and sand-to-binder (s/b) ratios for all mixtures were fixed at 0.5 and 3, respectively. A 20 % substitution rate for Portland cement, performed by mass, was chosen, based on the optimal results from previous studies [37–42]. The composition of the mortar mixtures is summarized in Table 2.

The specimens were cured in a moisture chamber at 20°C for 28 days before testing. The compressive strength test was conducted according to ISO 679:2009 [36] on six specimens of each mortar type. For the graphs, the average values of six measurements, along with the standard deviation, were used.

3.2. Capillary absorption

The capillary absorption measurement was conducted in accordance with the EN 480–5 standard [43]. Three specimens of each mortar type were prepared for testing. After 28 days of curing, the specimens were dried at 50°C until they reached a constant mass. The specimens were then weighed to determine their mass before adsorption. Each specimen was immersed in water to a depth of 5 mm with a 40 × 40 mm edge resting on a support, ensuring this edge was freely exposed to water. The container was then sealed with a lid in order to prevent evaporation.

Measurements were taken at the following time intervals: 0.25, 0.5, 1, 2, 4, 6, 24, 48, and 168 hours. At each interval, the specimens were

removed from the water, blotted with a paper napkin and weighed to determine the mass with absorbed water. The water level in the container was consistently maintained at a height of 5 mm.

The capillary absorption (C_a) in g/mm² for each time period was calculated using the formula:

$$C_a = \frac{M - M_0}{1600}, \quad (1)$$

where M_0 is the mass of the dry specimen in g, M is the mass of the specimen with absorbed water in g and 1600 is the surface of the exposed edge in mm².

For the graphs, the average values of three measurements, along with the standard deviation, were used.

3.3. Drying shrinkage

The drying shrinkage test was conducted following the EN 12617–4 standard [44] on three specimens of each mortar type, with modifications to the measurement duration and frequency. Cement mortar samples were poured into 40x40x160 mm moulds equipped with integrated attachment pins at both ends. After 24 hours, the specimens were removed from the moulds and placed on the ball-bearing rollers of a Shrinkage Measuring Device Type C from Testing Bluhm & Feuerherdt GmbH (Fig. 1). Shrinkage measurements were taken using a digital displacement gauge with a conical contact tip, featuring LVDT

**Fig. 1.** Drying shrinkage measurement device.**Table 2**

Composition of mortar mixtures (values in grams).

Mix	CEM I	CS105	MS105	WM105	CS500	MS500	WM500	Sand	Water
REF	450	0	0	0	0	0	0	1350	225
CS105	360	90	0	0	0	0	0	1350	225
MS105	360	0	90	0	0	0	0	1350	225
WM105	360	0	0	90	0	0	0	1350	225
CS500	360	0	0	0	90	0	0	1350	225
MS500	360	0	0	0	0	90	0	1350	225
WM500	360	0	0	0	0	0	90	1350	225

displacement sensors (measuring range: 5 mm, resolution: 0.31 μm , accuracy: 0.2 μm). The gauge was positioned on one end of the specimen to monitor time-dependent volume contraction by measuring changes in length. The opposite end was horizontally constrained by a fixed pin. Measurements were recorded every 2 hours for a duration of 90 days using a Schleibinger Geräte signal processor. Additionally, the environmental conditions, including temperature and relative humidity, were continuously monitored throughout the test to ensure accurate data collection.

3.4. Freeze-thaw resistance

Currently, there is no standardized procedure for evaluating the freeze-thaw resistance of mortar specimens. Therefore, a custom methodology was implemented. To measure freeze-thaw resistance, 40x40x160 mm specimens were dried at 50°C to a constant weight after 28 days of curing. The initial mass of each dried specimen was recorded. The specimens were then immersed in water in covered containers to prevent evaporation. These containers were placed in a freezer, cooled to -20°C at a rate of 4 °C/min and maintained at this temperature for 11 hours and 50 minutes. Subsequently, the specimens were heated to +20°C at the same rate. Each freeze-thaw cycle lasted 24 hours, with the temperature regime diagram for one cycle provided in Fig. 2.

The specimens underwent 28 freeze-thaw cycles. The selection of 28 freeze-thaw cycles aligns with previous studies assessing cementitious materials and provides a reliable comparison of different mortar formulations within a practical experimental timeframe. After completing the cycles, the specimens were dried again to a constant weight, and their final mass was recorded. The compressive strength of the specimens was then tested. Conclusions regarding the freeze-thaw resistance of the mortar specimens were drawn based on the percentage change in their mass and strength. For the analysis, the mean values of six measurements, along with the standard deviation, were used.

3.5. Fire resistance

Since no standardized test exists for assessing the fire resistance of mortar specimens, a custom test procedure was developed. Prior to exposure to elevated temperatures, the 40x40x160 mm mortar specimens were dried at 50°C until a constant mass was reached, at which point their initial mass was recorded. Subsequently, the samples were subjected to firing in a muffle furnace at target temperatures of 200°C, 300°C, 500°C, and 900°C for a duration of 2 hours. The heating rate was maintained at 10 °C/min, while the cooling was passive in the furnace. After cooling the specimens were weighed to determine the mass loss and then subjected to compressive strength tests to assess mechanical integrity. The fire resistance of the mortar specimens was quantified by calculating the percentage change in both mass and compressive strength. For the analysis, the mean values of six measurements, along with the standard deviation, were used.

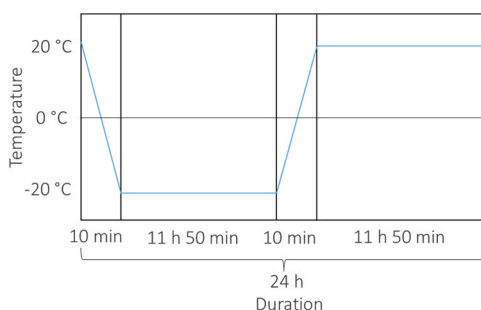


Fig. 2. Temperature regime of one freeze-thaw cycle.

3.6. Sulfuric acid resistance

As no established standards exist for evaluating the sulfuric acid resistance of mortar specimens, a custom procedure was employed. The resistance to the corrosive effect of sulfuric acid was assessed using 40x40x160 mm specimens after 28 days of curing. Initially, the specimens were dried at 50°C until reaching a constant mass, and their initial mass was recorded. The specimens were then immersed in plastic containers containing a 1 % (0.1 M) H_2SO_4 solution for 90 days. To prevent neutralization, the acid solution was renewed every 30 days.

After the 90-day immersion period, the specimens were rinsed with tap water and dried again at 50°C to a constant weight. The final mass and compressive strength of the corroded specimens were measured. The rate of sulfuric acid corrosion resistance of the mortars was assessed by expressing the mass and strength change as percentages. For the analysis, the mean values of six measurements, along with the standard deviation, were used.

3.7. Statistical analysis

Analysis of Variance (ANOVA) is a statistical tool used to analyse the collected data. In this research, ANOVA was employed to determine if there are statistically significant influences of different parameters on the durability features of the mortar specimens. The R programming language was used for data analysis. The factors included waste type, thermal treatment of waste powders, capillary adsorption and shrinkage. These factors were analysed in relation to the following responses: freeze-thaw resistance, fire resistance and sulfuric acid corrosion resistance. The analysis was organized in this way to comprehensively assess the durability of the cementitious material under various environmental conditions. Freeze-thaw resistance, fire resistance, and sulfuric acid resistance were chosen as dependent variables because they represent key durability challenges for cement-based materials in real-world applications. The four independent factors: waste type, thermal treatment of waste, capillary adsorption, and shrinkage, were selected because they are expected to significantly influence these durability properties. Waste type and thermal treatment affect the microstructure and chemical composition of the material, while capillary adsorption and shrinkage impact its porosity and mechanical stability.

The statistical significance of each factor's effect on durability properties was determined using the p-value. A p-value represents the probability of making an error when rejecting the null hypothesis, which assumes that the variable is not dependent on the factor. Thus, the smaller the p-value, the stronger the evidence of a relationship. In this study, a significance level of 0.05 was chosen, which is a widely accepted threshold in engineering and material science research. A stricter threshold could reduce Type I errors but might overlook meaningful effects due to increased Type II errors. Conversely, a more lenient threshold would increase the risk of false positives. If a p-value is below 0.05, there is strong evidence to reject the null hypothesis and conclude that the factor has a statistically significant influence on the durability property. Conversely, p-values above 0.05 suggest that the factor's effect is not statistically significant, implying that any observed variations may be due to random chance rather than a true relationship.

4. Results and discussion

4.1. Compressive strength

Fig. 3 presents the compressive strength values of the tested mortar specimens after 28 days of curing, compared to the reference Portland cement mortar. All investigated materials exhibited lower mechanical properties than the reference specimen (60.2 MPa). However, thermal treatment resulted in a slight improvement in the strength of the mortars. Thus, the compressive strength values for the mortars containing

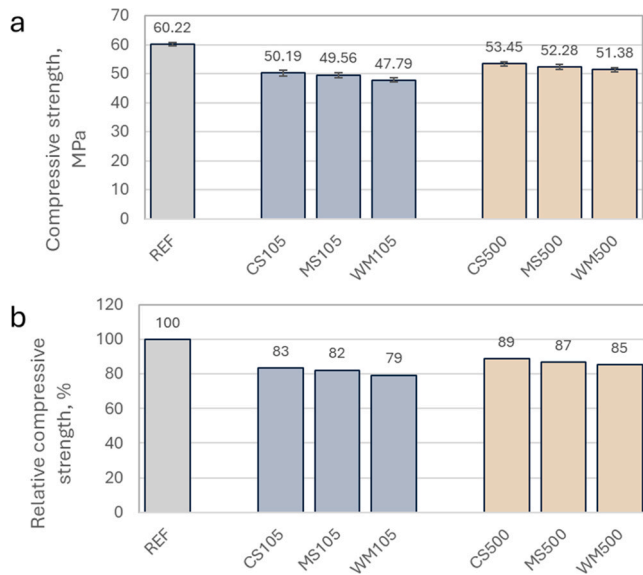


Fig. 3. Compressive strength of mortars containing 20 % demolition waste powders after 28 days of curing: (a) absolute values; (b) relative values.

CS105, MS105, and WM105 were 50.2 MPa, 49.6 MPa, and 47.8 MPa respectively, while for the mortars with CS500, MS500, and WM500, these values were 53.5 MPa, 52.3 MPa, and 51.4 MPa, respectively.

Nevertheless, all samples met the strength requirement set by the European standard EN 197-1 [45] for blended cement, corresponding to a strength class of 42.5 MPa. A more detailed discussion of the effect of thermally treated demolition waste powders on the strength of cement mortar can be found in the previous study [34]. In the current study, the compressive strength values after 28 days of curing are used to compare the strength loss of the mortars after the durability tests.

4.2. Capillary absorption

Fig. 4 presents the capillary water absorption curves for the investigated mortar specimens. The reference specimen, which contained no waste powders, exhibited the lowest absorption throughout the measurement period. The thermal activation of demolition waste powders

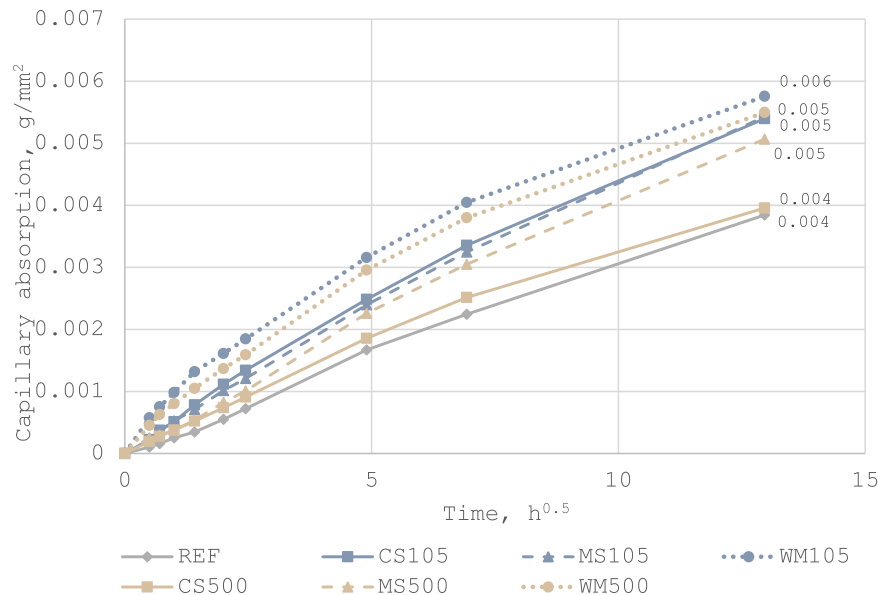


Fig. 4. Capillary absorption of mortars containing 20 % demolition waste powders.

resulted in reduced capillary absorption in all specimens. Notably, the mortar containing 20 % CS500 showed a similar absorption level to the reference specimen with a value of 0.004 g/mm², while without thermal activation this value was 0.005 g/mm². This reduction can be attributed to the increased reactivity of the thermally activated recycled powders, leading to matrix densification and reduced porosity. However, thermal activation had a smaller effect on the capillary absorption of specimens containing MS and WM, remaining at a level of around 0.005 g/mm² for all specimens. This can be explained by the lower content of hydrated cement in these samples compared to CS.

Fig. 5 compares absorption values after 7 days. It can be seen that the capillary absorption results align with the compressive strength test outcomes. Higher absorption corresponds to higher porosity, which in turn explains the lower mechanical strength observed in these specimens. These findings are consistent with the observations of Sun et al. [26] and Carriço et al. [15], who reported an increase in water absorption in cement mortar specimens with higher demolition waste powder content that correlated with compressive strength. They attributed this to the increased open porosity caused by the inherent porosity of the recycled concrete particles.

4.3. Drying shrinkage

Fig. 6 presents the drying shrinkage curves of cement mortar specimens over a 90-day period. The drying shrinkage of all specimens

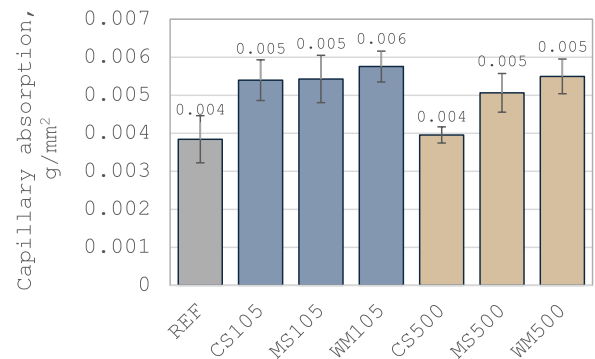


Fig. 5. Capillary absorption of mortars containing 20 % demolition waste powders after 7 days of contact with water.

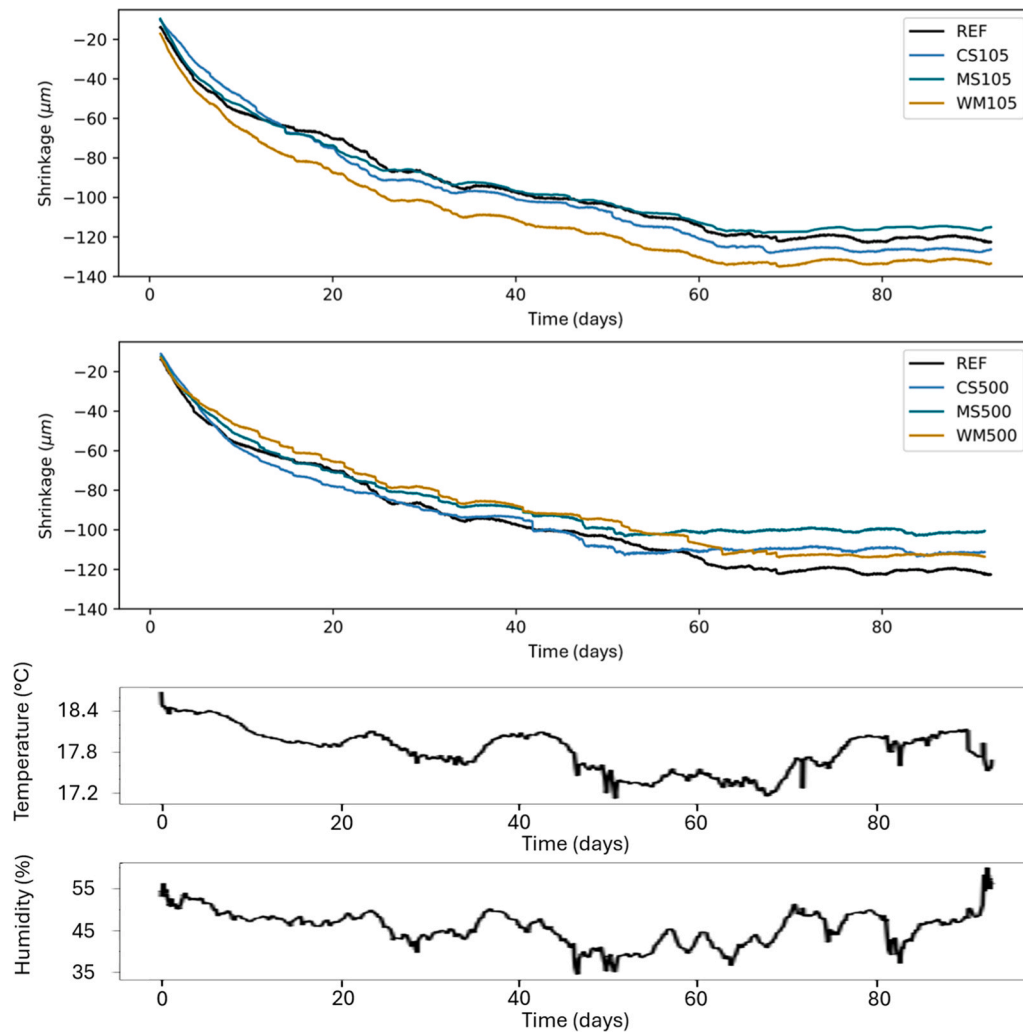


Fig. 6. Drying shrinkage of mortars containing 20 % demolition waste powders.

increased rapidly during the first 10 days, followed by a slower rate of increase over the next two months, ultimately stabilizing. The data indicate that specimens containing thermally activated powders exhibited lower shrinkage compared to those with non-calcined powders. Among the specimens, those containing MS demonstrated the lowest final shrinkage, even surpassing the reference sample, both before and after thermal activation. This observation aligns with findings from other researchers, who reported reduced drying shrinkage in specimens containing ceramic particles due to their pozzolanic activity [46,47].

At 90 days, the shrinkage values for CS105, MS105, and WM105 were 126 $\mu\text{m/m}$, 115 $\mu\text{m/m}$, and 134 $\mu\text{m/m}$, respectively. For CS500, MS500, and WM500, the values were 112 $\mu\text{m/m}$, 100 $\mu\text{m/m}$, and 112 $\mu\text{m/m}$, respectively. This reduction in shrinkage in mortars with thermally treated powders can be attributed to microstructural improvements resulting from the increased reactivity of the SCMs and the removal of organic impurities, as reported previously [34].

Interestingly, the REF specimens, despite lower capillary adsorption, exhibited higher drying shrinkage than those with thermally treated waste powders. Other researchers have found that drying shrinkage is more strongly related to pore size than to total pore volume. Zhang et al. [48] demonstrated that the smaller the pore diameter in a cementitious material, the higher its drying shrinkage. According to Schiller et al. [49], gel pores between C-S-H layers, which have no effect on capillary absorption in cement materials, play the primary role in drying shrinkage. This suggests that incorporating partially dehydrated

recycled cement containing C-S-H dehydration products different from Portland cement phases will affect shrinkage behaviour due to different morphology. Moreover, in a prior study on the investigated materials [34], it was shown that hydration products form within the larger pores of CS500, MS500, and WM500 samples, which may also contribute to reduced shrinkage in samples containing thermally activated powders.

4.4. Freeze-thaw resistance

The freeze-thaw resistance of cement mortar specimens was assessed by evaluating the mass loss (Fig. 7) and compressive strength loss (Fig. 8) after repeated freezing and thawing cycles. The results indicate that, overall, thermal activation of recycled powders does not significantly enhance the freeze-thaw resistance of the mortars. The mass loss was $1.07 \pm 0.02 \%$ for REF, $1.28 \pm 0.05 \%$, $0.93 \pm 0.12 \%$ and $1.04 \pm 0.00 \%$ for CS105, MS105 and WM105, respectively, and $1.34 \pm 0.08 \%$, $1.28 \pm 0.03 \%$ and $1.42 \pm 0.24 \%$ for CS500, MS500 and WM500, respectively. The strength loss values were $8.49 \pm 2.27 \%$, $4.38 \pm 2.09 \%$ and $5.25 \pm 1.63 \%$ for specimens containing 20 % of CS105, MS105 and WM105, respectively, while for samples incorporating CS500, MS500 and WM500, the strength loss values were $6.03 \pm 2.45 \%$, $4.72 \pm 1.88 \%$ and $6.47 \pm 3.13 \%$, respectively.

A correlation between mass loss and strength loss is observed, as higher material degradation due to freeze-thaw cycles leads to a greater reduction in mechanical integrity. This relationship arises because mass loss results from surface scaling and microstructural damage, which in

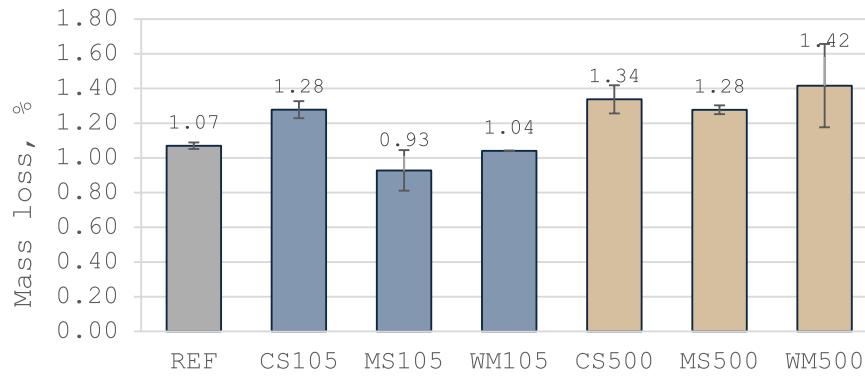


Fig. 7. Mass loss of mortars containing 20 % demolition waste powders after 28 freeze-thaw cycles.

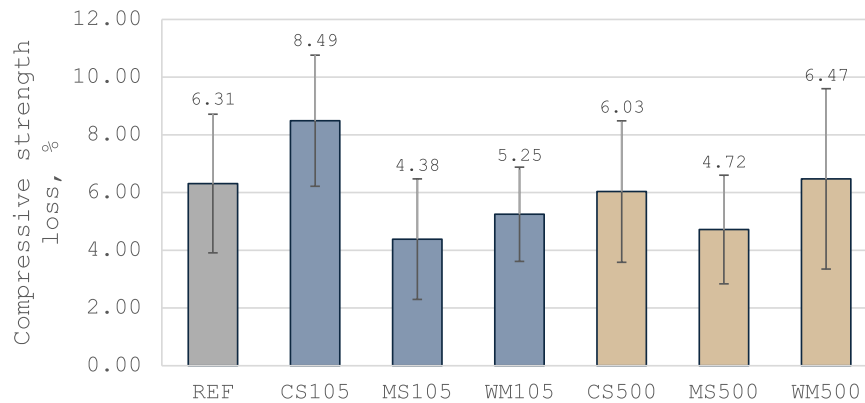


Fig. 8. Compressive strength loss of mortars containing 20 % demolition waste powders after 28 freeze-thaw cycles.

turn compromises the mortar's ability to bear loads. However, the variations in mass and strength loss among different specimens suggest that the composition of recycled powders influences the extent of deterioration.

Considering the associated measurement errors, it can be concluded that the freeze-thaw resistance of all specimens is comparable to that of the reference mortar, which exhibited a strength loss of 6.31 ± 2.40 %. Specimens containing MS105 and MS500 demonstrated slightly lower mass and strength losses after 28 freeze-thaw cycles, suggesting a potential positive influence of pozzolanic ceramic particles on the frost resistance of cement mortars.

Although freeze-thaw resistance is influenced by the open porosity of

cement mortars, here no clear correlation is observed between the capillary absorption behavior and the observed mass and strength losses. This suggests the involvement of additional parameters that significantly affect the frost resistance of the materials. As highlighted by Rhardane et al. [50], freeze-thaw resistance is also influenced by factors such as presence of ions in the pore solution and the composition of the cement paste. Furthermore, the pore size distribution plays a critical role, with larger pores contributing to improved freeze-thaw resistance by providing space for water expansion during freezing [51,52]. As shown in Fig. 9, specimens MS105 and MS500, which exhibited the highest freeze-thaw resistance, contain the greatest number of macropores. Additionally, the thermal treatment of waste powders results in

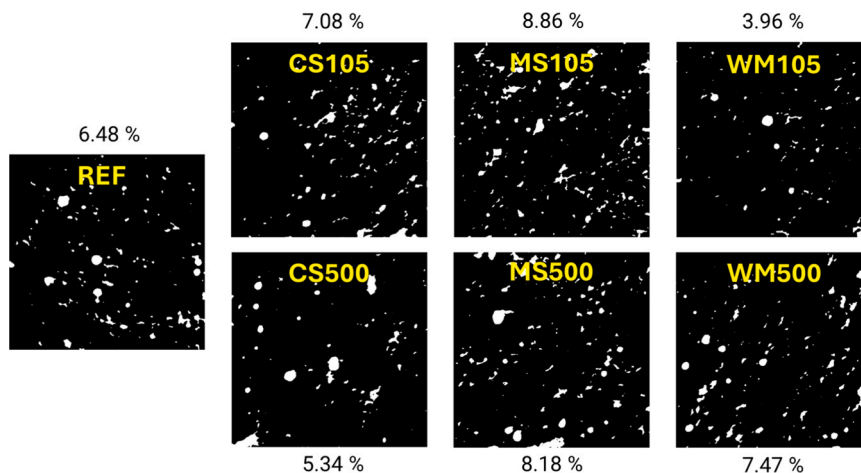


Fig. 9. Contrast-enhanced macropore distribution in mortars cross-sections.

pore enlargement.

4.5. Fire resistance

Fig. 10 illustrates the mass loss of the specimens after exposure to 200°C, 300°C, 500°C, and 900°C. At 200°C, all specimens exhibited a mass loss of less than 1 %, mainly attributed to the decomposition of gypsum, ettringite, and partial degradation of the C-S-H gel, leading to water release. At 300°C, the continued decomposition of the C-S-H gel resulted in an approximate mass loss of 1 % across all mortars. When fired at 500°C, the decomposition of the C-S-H gel persisted, accompanied by the initiation of portlandite decomposition, also involving water removal. At this stage, the specimens lost between 2.2 % and 2.6 % of their mass. Finally, at 900°C, the complete decomposition of both C-S-H gel and portlandite occurred, together with the decomposition of calcite with release of carbon dioxide. The mass loss at this temperature ranged from 4.6 % to 5.0 %. Overall, no significant differences were observed in the mass loss between the different mortar specimens.

Fig. 11 presents the compressive strength loss of the mortar specimens after exposure to 200°C, 300°C, 500°C, and 900°C. At 200°C, the change in strength was clearly dependent on the type of waste used in the cement mortar. Thus, the strength loss of specimens containing CS105 and CS500 was 7.8 % and 3.9 %, respectively, while specimens containing WM105 and WM500 showed strength gains of 5.6 % and 14.4 %, respectively. At 300°C, the strength of WM500-containing specimens increased by 6.0 %, while all other specimens experienced a loss in strength ranging from 10.0 % for MS500 to 22.0 % for CS105. The strength gain in WM-containing specimens can be attributed to autoclave processes which forms calcium silicates and aluminates due to the higher content of pozzolanic oxides in WM (Table 1). In addition, WM powders contained fewer sulfates, which are prone to decomposition at these temperatures compared to MS.

At 500°C, all specimens exhibited a compression strength loss. Mortars containing WM105 and WM500 had the lowest compression strength losses of 27.9 % and 19.8 % respectively, indicating better performance. Other specimens experienced strength losses ranging from 31.8 % to 35.2 %. At this temperature, visible cracks appeared on the surfaces of all mortar bars. After exposure to 900°C, almost all compressive strength was lost, with strength losses ranging from 89.0 % for WM105 to 92.2 % for CS105. The surfaces of the specimens were riddled with cracks, and white inclusions of free lime formed by the decomposition of portlandite and calcite were visible.

While mass loss is mainly associated with the decomposition of hydration products and the release of chemically bound water and carbon dioxide, strength loss is more strongly influenced by microstructural changes, crack formation, and phase transformations. The differences in strength loss between mortars, despite similar mass loss values, suggest that the composition of waste materials plays a significant role in determining mechanical stability at elevated temperatures, particularly

through pozzolanic activity, sulfate content, and the formation of new cementitious phases during heating.

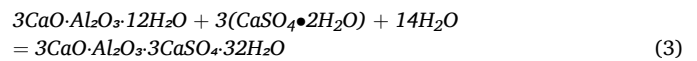
Specimens containing CS105 showed slightly lower fire resistance than the reference specimen. However, the results demonstrate an improved fire resistance in WM-containing mortars. This improved performance can be explained by the chemical and mineralogical composition of the waste:

- WM and MS contain muscovite, a refractory mineral.
- WM has a lower calcite content.
- WM has the highest content of pozzolanic oxides, and a lower sulfate content compared to MS.

Additionally, a correlation between fire resistance (Fig. 11) and capillary absorption (Fig. 5) can be observed. Indeed, porous materials provide better thermal insulation due to pores and voids filled with air, which is poor conductor of heat. The coefficient of thermal expansion (CTE) of the wastes used also plays an important role in the fire resistance of cement mortars [53,54].

4.6. Sulfuric acid resistance

The resistance of mortar specimens to sulfuric acid corrosion was evaluated based on changes in mass (Fig. 12) and compressive strength loss (Fig. 13). Interestingly, all specimens gained mass after exposure, as also reported by Cao et al. [55]. The mass gain is primarily attributed to the reaction of calcium-containing compounds in cementitious materials with sulfate ions from sulfuric acid, forming gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) according to formulas (2) and (3). These compounds accumulate in the upper layer of the mortar, resulting in a mass increase.



The mass gain of specimens with recycled powders was comparable to the reference specimen (1.19 %), with CS105, MS105, and WM105 showing mass increases of 1.37 %, 1.51 %, and 1.45 %, respectively. Thermal treatment of the powders slightly reduced the weight gain, with CS500, MS500, and WM500 exhibiting gains of 1.18 %, 1.32 %, and 1.25 %, respectively. However, change in mass alone is not a reliable indicator of corrosion resistance because it reflects both the addition of sulfate (SO_4^{2-}) and hydrogen (H^+) ions and the leaching of calcium (Ca^{2+}) and aluminum (Al^{3+}) ions from the mortar, as highlighted by Cao et al. [54] and Khan et al. [56]. The extent of these processes also depends on the acid concentration and immersion duration. The higher the concentration of sulfuric acid and the longer the exposure time, the greater the leaching processes and the degradation of the cement material

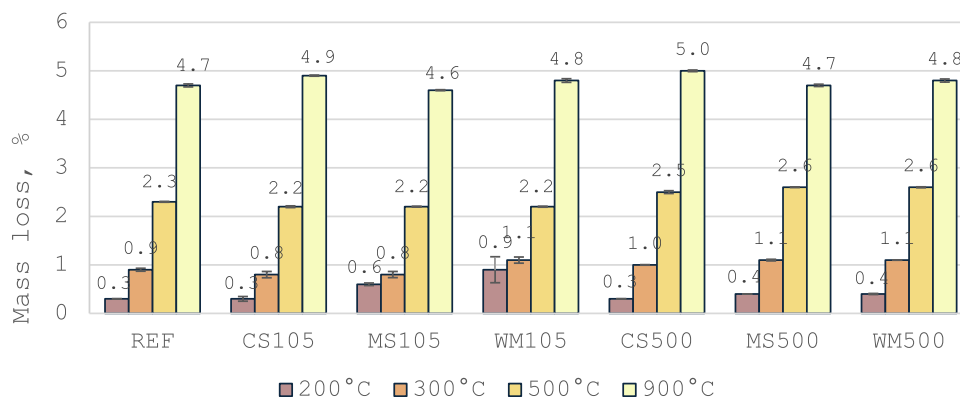


Fig. 10. Mass loss of mortars containing 20 % demolition waste powders after firing at 200°C, 300°C, 500°C and 900°C.

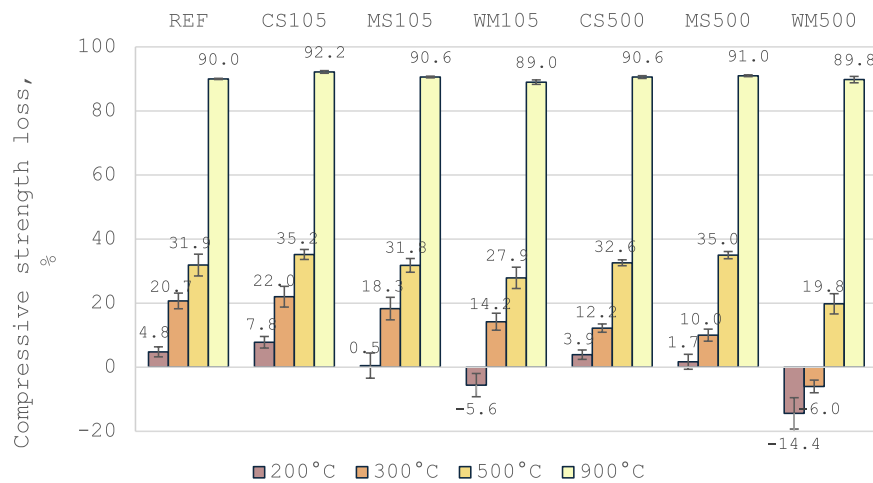


Fig. 11. Compressive strength loss of mortars containing 20 % demolition waste powders after firing at 200°C, 300°C, 500°C and 900°C.

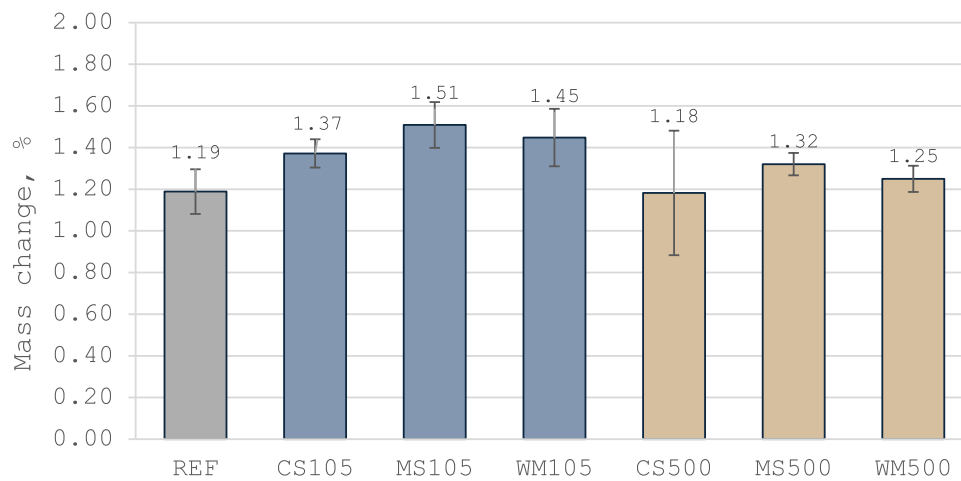


Fig. 12. Mass change of mortars containing 20 % demolition waste powders after exposure to sulfuric acid solution.

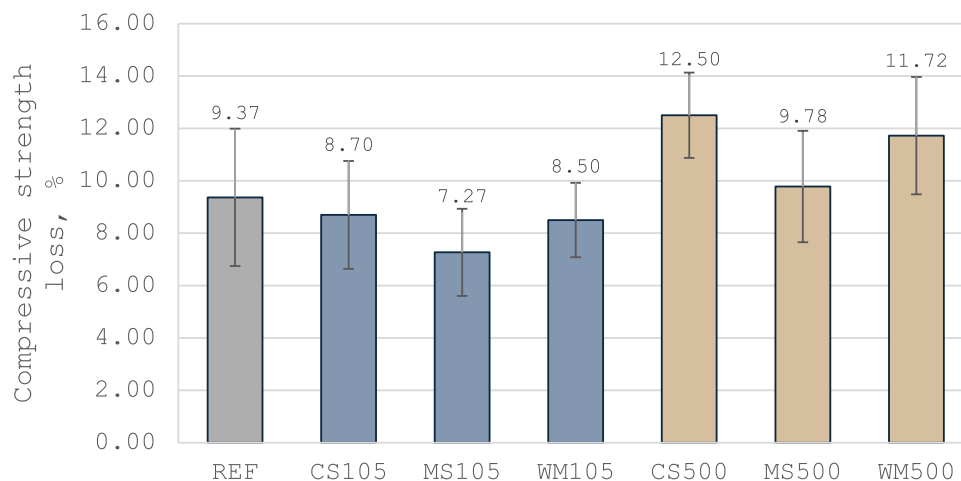


Fig. 13. Compressive strength loss of mortars containing 20 % demolition waste powders after exposure to sulfuric acid solution.

[57–59].

Compressive strength loss provided a clearer picture of the acid resistance of the mortars. Specimens containing uncalcined waste powders exhibited slightly lower strength loss than the reference mortar (9.37 %), with CS105, MS105, and WM105 showing losses of 8.70 %, 7.27 %, and 8.50 %, respectively.

In contrast, thermal treatment increased the sensitivity of the mortars to acid attack, resulting in higher strength losses for CS500, MS500, and WM500 (12.50 %, 9.78 %, and 11.72 %, respectively). This heightened sensitivity is likely due to increased reactivity of the reactivated powders combined with the

relatively higher porosity of the mortars and the formation of additional C-S-H gel during hydration, as reported in prior study [34].

Interestingly, an inverse relationship between mass gain and strength loss was observed, with specimens exhibiting higher mass gain tending to retain more compressive strength. This can be attributed to the formation of gypsum and ettringite, which, despite their expansive nature, partially fill voids in the upper layers and temporarily limit further acid penetration into the material.

Among all specimens, those containing MS powders showed the lowest loss of strength despite having the highest mass gain. This behavior can be attributed to the chemical inertness of the ceramic particles in MS mortars. Ceramic particles, consisting of crystalline or semi-crystalline aluminosilicates, are highly stable in acidic environments, unlike more reactive cement phases such as calcium hydroxide and C-S-H gel. The inert ceramic particles resist dissolution when exposed to sulfuric acid, thereby minimizing internal degradation.

Thus, the acid reacts with calcium-bearing phases in the surrounding cement paste, leading to localized formation of gypsum and ettringite near the surface or in voids. These expansive reaction products increase the specimen's mass due to their high molecular weight and cause the structural damage of the surface layers. The presence of ceramic particles in the cement matrix reduces overall the reactivity and improves the resistance to acid penetration. This explains why MS mortars exhibit superior performance under acidic conditions, with lower strength loss and mass changes compared to other specimens.

4.7. Statistical analysis

The statistical approach of multifactor Analysis of Variance (ANOVA) helps in examining the relationships between multiple factors and dependent variables. In this study, ANOVA was applied to evaluate how freeze-thaw resistance, fire resistance and resistance to H_2SO_4 corrosion were influenced by four independent factors: waste type, thermal treatment of waste, capillary adsorption and shrinkage. This method is particularly advantageous for assessing differences across multiple groups and for minimizing the influence of random errors. The

Table 3
ANOVA results.

Dependent Variable	Independent Factor	Sum Sq ^a	Mean Sq ^b	F-value ^c	p-value ^d
Freeze-thaw resistance	Waste type	25.07	12.53	4.92	0.033
	Thermal treatment	0.32	0.32	0.13	0.732
	Capillary adsorption	19.42	19.42	7.62	0.020
	Shrinkage	4.91	4.91	1.93	0.195
Fire resistance	Waste type	$4.88 \cdot 10^{11}$	$2.44 \cdot 10^{11}$	12.91	0.002
	Thermal treatment	$8.83 \cdot 10^8$	$8.83 \cdot 10^8$	0.05	0.833
	Capillary adsorption	$3.31 \cdot 10^{11}$	$3.31 \cdot 10^{11}$	17.51	0.002
	Shrinkage	$1.30 \cdot 10^9$	$1.30 \cdot 10^9$	0.07	0.798
H_2SO_4 resistance	Waste type	16.37	8.18	3.91	0.056
	Thermal treatment	40.68	40.68	19.43	0.001
	Capillary adsorption	1.22	1.22	0.58	0.463
	Shrinkage	7.81	7.81	3.73	0.082

^a Sum Sq (Sum of Squares) – the total variation in the dependent variable explained by each independent factor.

^b Mean Sq (Mean Square) – the Sum of Squares divided by the degrees of freedom, representing the average variation caused by each factor.

^c F-value – the ratio of the Mean Square of the factor to the Mean Square of the residual error, used to determine statistical significance.

^d p-value – the probability of obtaining the observed results under the null hypothesis.

results of statistical analysis, conducted using R software, are presented in Table 3. The p-values highlight the degree of significance for each factor's effect on durability properties, with values below 0.05 indicating statistically significant dependencies.

After conducting ANOVA to identify significant differences among group means (waste types), Tukey's post hoc test (Table 4) was employed to determine which specific group pairs differed significantly. While ANOVA identifies whether there is a significant effect of a factor overall, it does not indicate which groups differ from each other. This test controls for Type I error across multiple comparisons by adjusting p-values, ensuring robust and reliable results.

For freeze-thaw resistance, both factors, the waste type ($p = 0.033$) and the capillary adsorption ($p = 0.020$), exhibited statistically significant effects, while the factor thermal treatment ($p = 0.732$) had no statistically significant impact. Post hoc analysis further identified a significant difference between MS and CS ($p = 0.026$), suggesting that the freeze-thaw resistance of MS-based specimens is superior. However, no significant differences were observed between WM and the other types.

Similarly, for fire resistance, waste type ($p = 0.002$) and capillary adsorption ($p = 0.002$) demonstrated highly significant effects, while thermal treatment ($p = 0.833$) had no statistically significant impact. Post hoc analysis revealed that CS exhibited significantly lower fire resistance compared to MS ($p = 0.009$) and WM ($p = 0.002$), while no significant difference was observed between WM and MS.

In terms of resistance to H_2SO_4 corrosion, thermal treatment ($p = 0.001$) was found to have a highly significant influence, while waste type ($p = 0.056$) showed a near-significant trend. Capillary adsorption ($p = 0.463$) did not exhibit a statistically significant effect. Tukey post hoc comparisons showed no significant differences between the waste types, although MS and CS approached significance ($p = 0.055$), indicating a potential difference.

Shrinkage did not exhibit a statistically significant effect on any of the analyzed durability parameters. The dependence on drying shrinkage was not revealed in this experiment, likely because the mechanisms of resistance to temperature changes and acids are complex and depend on more factors than just microcracks. Other parameters, such as differences in hydration products and macroporosity, appear to play a greater role in influencing durability under the conditions tested. It is possible that with longer test durations, the influence of shrinkage-related cracking would become more apparent.

5. Conclusions

The study investigated the effects of incorporating thermally activated and non-activated demolition wastes as SCMs in cement mortars on durability properties, including freeze-thaw resistance, sulfuric acid resistance and fire resistance. Based on the findings, the following conclusions can be drawn:

1. The reference specimen showed the lowest capillary absorption, while thermal activation reduced this parameter in all specimens, with a smaller effect observed in those containing MS and WM compared to CS.
2. Thermal activation of waste powders reduced the drying shrinkage of mortar specimens. The REF specimen exhibited higher drying shrinkage compared to those containing thermally activated waste powders, likely due to the presence of gel pores in the C-S-H gel matrix and the growth of hydration products in the larger pores of mortars with rehydrated waste powders.
3. Thermal activation of recycled powders had no significant effect on the frost resistance of mortars, whereas the type of waste material did. No visually clear correlation was observed between capillary absorption and freeze-thaw resistance, suggesting the involvement of additional influencing factors. However, statistical analysis confirmed a significant dependence of freeze-thaw resistance on both

Table 4

Tukey post hoc test results.

Dependent Variable	Comparison	Difference ^a	Lower CI ^b	Upper CI ^c	p-value ^d
Freeze-thaw resistance	MS – CS	−2.89	−5.41	−0.36	0.026
	WM – CS	−1.54	−4.07	0.98	0.262
	WM – MS	1.35	−1.18	3.87	0.350
Fire resistance	MS – CS	−298864.2	−516467.2	−81261.3	0.009
	WM – CS	−383975.7	−601578.6	−166372.8	0.002
	WM – MS	−85111.5	−302714.4	132491.5	0.551
H ₂ SO ₄ resistance	MS – CS	−2.24	−4.53	0.05	0.055
	WM – CS	−0.55	−2.84	1.74	0.794
	WM – MS	1.69	−0.60	3.98	0.156

^a Difference – the mean difference between the two groups.^b Lower CI (Lower Confidence Interval) – the lower bound of the confidence interval for a 95 % confidence level.^c Upper CI (Upper Confidence Interval) – the upper bound of the confidence interval for a 95 % confidence level.^d p-value – the probability of observing the obtained results under the null hypothesis.

the waste type and capillary absorption, while thermal treatment showed no statistically significant impact.

4. Fire resistance was strongly influenced by the type of waste used in the cement mortar and porosity, but it was not affected by the thermal treatment of the waste. Mortars containing WM exhibited the highest fire resistance, attributed to the chemical and mineralogical composition of the waste.
5. Thermal activation of waste powders reduced the sulfuric acid corrosion resistance of the mortars due to the increased reactivity of the activated powders.

Overall, the findings demonstrate that the type of waste has a significant impact on the durability properties of cement mortars. Specimens containing ceramic particles consistently outperformed those with pure concrete waste and even the reference mortar, underscoring their potential as a sustainable alternative in cementitious materials production. The influence of capillary absorption on both freeze-thaw and fire resistance highlights the critical role of porosity and air voids in improving thermal insulation. However, thermal treatment of the wastes did not improve the durability of the mortars and, in the case of sulfuric acid resistance, actually reduced it.

While this research does not delve into the detailed processes occurring under the influence of aggressive environmental factors, it provides valuable insights into the potential durability of cement materials containing different CDWs. However, a more detailed investigation into the mineralogical and microstructural changes during heating, cooling and acid expose is required for a definitive explanation of the research findings.

CRediT authorship contribution statement

Tokareva Anna: Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Waldmann Danièle:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization.

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.

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Data Availability

The data that has been used is confidential.

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