J Sustain Const Mater Technol, Vol. 8, Issue. 4, pp. 307–318, December 2023

Journal of Sustainable Construction Materials and Technologies
Web page info: https://jscmt.yildiz.edu.tr
DOI: 10.47481/jscmt.1361326

Research Article

Mechanical, freeze-thaw, and sorptivity properties of mortars prepared with different cement types and waste marble powder

Rümeysa GÜRGÖZE[1], Zinnur ÇELİK*[2], Ahmet Ferhat BİNGÖL[1]

[1]*Department of Civil Engineering, Atatürk University Faculty of Engineering, Erzurum, Türkiye*
[2]*Pasinler Vocational School, Atatürk University, Erzurum, Türkiye*

ARTICLE INFO

Article history
Received: 16 September 2023
Revised: 31 October 2023
Accepted: 31 October 2023

Key words:
Calcium aluminate cement, freeze-thaw, mechanical properties, waste marble powder

ABSTRACT

The cement production process contributes significantly to CO_2 gas emissions and environmental pollution. To reduce this adverse effect, the substitution of waste marble powder as a cement additive was investigated. In this study, the properties of mortar specimens were analyzed by using waste marble powder as a partial substitute for three different cement types: CEM I 42.5R Ordinary Portland Cement (OPC), CEM II/B-L 42.5R White Cement (WC) and CA-40 Calcium Aluminate Cement (CAC). Waste marble powder has been replaced with cement at 5%, 10%, and 15%. The compressive and flexural strength, capillary water absorption, and sorptivity values of the prepared mixtures were determined before and after freezing and thawing. It was carried out after 28 days of water curing on 50 x 50 x 50 mm specimens for compressive strength and 160 x 40 x 40 mm specimens for flexural strength test. Freeze-thaw testing of the mixture samples was conducted according to ASTM C666 Procedure A. Test results showed that the highest compressive strength before freeze-thaw was obtained in calcium aluminate cement-based mortars containing 10% by weight waste marble powder replacement for cement. The appropriate waste marble powder ratio was determined as 10% in all cement types used in the study. Before freeze-thaw, the mechanical properties of CAC-based mixtures were higher than those of other cement types. However, as the number of freeze-thaw cycles increased, the strength losses were more significant compared to OPC and WC.

Cite this article as: Gürgöze, R., Çelik, Z., & Bingöl A. F. (2023). Mechanical, freeze-thaw, and sorptivity properties of mortars prepared with different cement types and waste marble powder. *J Sustain Const Mater Technol*, 8(4), 307–318.

1. INTRODUCTION

Concrete is the most widely used building material worldwide due to its low price, easy accessibility of its components, ability to give the desired shape, and provide the necessary strength and durability [1, 2]. Cement, the most used binder material in concrete production, is the primary source of CO_2 emissions. Ordinary Portland Cement (OPC) production contributes about 5% to 7% of global CO_2 emissions [3, 4]. It has been determined that the concrete production in Türkiye as of 2014 is approximately 70 Million Tonnes (Mt); therefore, about 65 Mt of CO_2 has been released [5, 6].

Cement production significantly impacts CO_2 emissions and can be reduced using waste materials that improve concrete's fresh and mechanical properties. Among these waste materials, marble powder, mainly found in Turkey, India, China, and Italy, is used as a cement substitute in concrete production. The production of marble waste is estimated to be around 3 Mt annually [5]. During the processing of natural marble, a significant amount of powder particles are released into the environment, creating waste.

***Corresponding author.**
*E-mail address: zinnur.celik@atauni.edu.tr

Published by Yıldız Technical University Press, İstanbul, Türkiye

Disposing waste materials from these powder particles can be complex and contribute significantly to environmental damage by contaminating natural resources [6].

Marble powder is used in concrete and mortar production due to its chemical structure and filling properties. This can reduce environmental damage and contribute to a sustainable approach in the construction industry. The filling effect of marble powder forms a denser mixture, improving the transition zone and cement matrix. This may cause an increase in the strength of the mixtures by adding low amounts of marble powder. Additionally, dolomite ($CaMg(CO_3)_2$) found in WMP reacts with the alkalis of cement to form calcium carbonate ($CaCO_3$). As a result of the reaction between calcium carbonate and the C_3A component of cement, a more compact structure is formed that increases the binding capacity of the matrix [7]. Munir et al. [8] reported that calcite in WMP reacted with C_3A to form calcium carbo-aluminate, and better compressive strength was obtained in mixtures containing WMP. There are several studies in the literature on the substitution of waste marble powder (WMP) for Ordinary Portland Cement (OPC). Uysal and Yılmaz [9] determined that the properties of fresh concrete were improved by replacing the cement with marble powder at 10%, 20%, and 30% by mass. Aliabdo et al. [10] determined that the 28-day compressive strength decreased by 7%, 4%, 5%, and 14% when replacing 5%, 7.5%, 10%, and 15% marble powder with OPC compared to the control specimen. Ergün [11] reported that substituting 5% and 7.5% WMP for cement increased the compressive strength, but using 15% decreased the strength. Ashish [5] observed that adding 15% WMP to the concrete increased the compressive strength by 4.5% and 10.4% at 28 and 91 days of curing times, respectively, compared to the control specimen. Rodrigues et al. [12] determined that replacing cement with marble powder up to 10% in concrete does not adversely affect the compressive strength. Still, replacing 20% marble powder reduces the compressive strength by 25%.

Studies in the literature on the durability of concrete are attracting increasing attention. Freeze-thaw (FT) is among the main reasons for the loss of durability of concrete, especially in cold climates [13, 14]. Due to the porous structure of concrete, repetitive FT cycles cause the concrete to lose strength and crumble by exfoliating [15]. Freeze-thaw studies in the literature on waste marble powder have generally focused on substituting WMP with fine aggregate in concrete [15–18]. İnce et al. [16] reported that the FT resistance of concrete to which waste marble powder was added increased.

Another issue is that the Ordinary Portland Cement (OPC) was used in all studies based on the literature above. However, due to the large amount of CO_2 released by OPC, studies on the discovery and application of cementing materials to ensure sustainability in cement production continue [19]. Calcium aluminate cement (CAC) releases approximately 30% less CO_2 than OPC during production. In addition, CAC is preferred in refractory material production, wastewater, and industrial floor applications due to its high early strength and resistance to harsh environmental conditions such as acid and high temperature [20, 21]. However, the use of CAC in structural system elements is prohibited due to instability in compressive strength due to the transformation of hydration products that contribute to the early high strength of CAC [22, 23].

Table 1. Chemical characteristics of OPC, CAC, WC, SF and WMP

Compound	OPC (%)	CAC (%)	WC (%)	SF (%)	WMP (%)
SiO_2	17.73	3.60	21.60	95.60	0.80
Al_2O^3	4.56	39.80	4.05	0.40	0.60
Fe_2O_3	3.07	17.10	0.26	0.80	0.80
CaO	62.81	36.20	63.70	0.40	57.20
MgO	2.07	0.65	1.30	0.60	9.60
K_2O	0.62		0.35	0.40	0.03
SO_3	2.90	0.04	3.30	0.20	0.04

2. RESEARCH SIGNIFICANCE

Studies on sustainability in the building sector have attracted much attention in recent decades. In most studies, WMP has been used by replacing fine aggregate. Although this situation contributes to the use of waste materials, it does not positively affect the CO_2 emission originating from the cement. In addition, most of the studies in the literature have focused on OPC. However, regarding sustainability, examining and using CAC, which causes less CO_2 emissions, is very valuable in reducing environmental problems. This study aims to investigate the freeze-thaw effect, lacking in the literature, by substituting waste marble powder in the mortar instead of cement. Another important aim is to examine the usability of CAC and white cement (WC) with WPM and compare it with OPC. The study was considered to compare CEM I cement, which is frequently used in practice, with two particular types of cement (white and calcium aluminate). The use of CAC and WMP together is important in terms of sustainability. In this context, mortar specimens were prepared by replacing 5%, 10%, and 15% WMP with three different types of cement. In addition, silica fume (SF) was used at the ratio of 10% by weight of cement in all mixtures. These specimens were subjected to compressive strength, flexural strength, and capillary permeability tests. In addition, the specimens were left to 3 different freeze-thaw cycles, and the same tests were repeated.

3. EXPERIMENTAL PROGRAMME

3.1. Materials

In this study, three different types of cement were used in the preparation of mortar mixes: OPC (CEM I 42.5 R), CAC (CA-40), and WC (CEM II/B-L 42.5R). WMP was replaced by weight with three different cement types at 5%, 10%, and 15%. In addition, since the risk of segregation was observed in the mixtures as a result of preliminary experiments, 10% of the binder amount of SF was used in all mixtures to increase the viscosity. The chemical and physical properties of OPC, CAC, WC, SF, and WMP are in Tables 1 and 2, respectively.

Table 2. Physical properties of OPC, CAC, WC, SF and WMP

Property	OPC	CAC	WC	SF	WMP
Specific gravity	3.15	3.25	3.06	2.20	2.73
Loss on ignition (%)	2.05	0.30	3.20	0.60	42.60
Insoluble residue (%)	0.66	0.16	0.18		0.91
Specific surface	3450[a]	3000[a]	4600[a]	19800[b]	
Compressive strength of cement (Mpa)					
Two days	25	60	24		
28 days	48		46		

a: Blaine method (cm^2/g); b: BET method (m^2/kg).

Table 3. Mix proportions of mortars (kg/m^3)

Mixture codes	Cement	Silica fume	WMP	Fine agg.	Water	Superplasticizer
OPC-0	540	60		1404	252	6
OPC-5	510	60	30	1398	252	6
OPC-10	480	60	60	1396	252	6
OPC-15	450	60	90	1391	252	6
CAC-0	540	60		1416	252	6
CAC-5	510	60	30	1412	252	6
CAC-10	480	60	60	1409	252	6
CAC-15	450	60	90	1404	252	6
WC-0	540	60		1391	252	6
WC-5	510	60	30	1386	252	6
WC-10	480	60	60	1383	252	6
WC-15	450	60	90	1380	252	6

Within the scope of the experimental study, the specific gravity and water absorption ratio of the sand were determined according to EN 1097-6 [24]. River sand with a specific gravity of 2.60, water absorption value of 1.80%, and fineness modulus of 3.42 was used as fine aggregate. In addition, in this study, a new generation superplasticizer polycarboxylate-based with the pH and specific gravity of 6 and 1.065, respectively.

3.2. Mix Proportions

The mixing ratios of the mortar specimens prepared within the scope of this study are given in Table 3. In similar studies in the literature, it has been determined that generally, between 500–600 kg/m^3 of binder material is used. In EFNARC 2005 [25], the total powder content was recommended to be 400–600 kg/m^3. All mixtures kept the binder amount constant at 600 kg/m^3 for these reasons. In addition, 10% of the binder amount of SF was used in all the mixtures. Waste marble powder was replaced with cement at 5%, 10%, and 15% of the total binder amount. The water/binder ratio was determined as 0.42 in all mixtures. To ensure the workability of the fresh mortar, 1% of the binder amount was used as a superplasticizer. According to these parameters, 13 different mortar mixtures were prepared. Each mixture is named according to the type of cement used in the mix and the ratio of waste marble powder. For example, in a CAC-10 code, the first three letters (CAC) indicate the use of calcium aluminate cement, and the adjacent numbers (10) represent the amount of waste marble powder in the mortar mix.

3.3. Test Methods

To determine the workability of the self-compacting mortar (SCM), the mini-slump flow test was applied according to EFNARC [25]. The workability values of the SCMs were evaluated according to criteria of 240–260 mm for the slump flow diameter. The mortar specimens' compressive and flexural strength tests were carried out by ASTM C109 [26] and ASTM C348 [27] standards, respectively. It was carried out after 28 days of water curing on 50 x 50 x 50 mm specimens for compressive strength and 160 x 40 x 40 mm specimens for flexural strength test.

Also, within the scope of the experimental program, 50 × 50 × 50 mm cube specimens were tested at age 28 days according to ASTM C1585-13 [28] to calculate the capillary water absorption and sorptivity coefficients of SCM mixtures. For the capillary water absorption test, the four side surfaces of the specimens were covered with a seal using vinyl electrician tape and exposed to 1–3 mm of water from only one surface. All mixture specimens' weight and cross-sectional area were measured before the capillary water absorption test. Capillary water absorption was deter-

Figure 1. SCM specimens **(a)** in mixture, **(b)** slump test, **(c)** in water curing pool, **(d)** sorptivity test, **(e)** flexural strength test.

mined by measuring the weights of the specimens at 5, 10, 30, 60, 120, 180, 240, 300, and 360 min time intervals. Laboratory images of experimental studies are given in Figure 1.

Freeze-thaw (F-T) testing of mixed specimens was performed according to ASTM C666 [29] Procedure A. The specimens were subjected to 3 different cycles (30, 60, 90) after 28 days of curing, and then compressive strength, flexural strength, and capillary permeability tests were performed. Ambient conditions were set to have cycles of freezing at -18°C and thawing at 4°C.

4. RESULTS AND DISCUSSIONS

4.1. Workability

The slump-flow diameter test results of the mixtures are presented in Figure 2. Significant decreases were detected in the slump flow diameters of the mixtures with WMP added, and this was more evident in the mixtures using 10% and 15% WMP. This can be attributed to the fact that the specific surface area of waste marble powder is higher than cement, thus reducing workability by increasing internal friction [5]. Rashwan et al. [30] reported that waste marble powder's angular, rough shape and high fineness reduce workability. Li et al. [31] stated that drier mixtures were obtained by substituting cement paste with waste marble powder at higher rates than the control mixture. It was determined that the flow diameters of all mixtures were between 240–260 mm, specified in EFNARC, with the effect of the superplasticizer. OPC-based mixtures were obtained with slump flow diameters of 258, 257, 252, and 248 mm

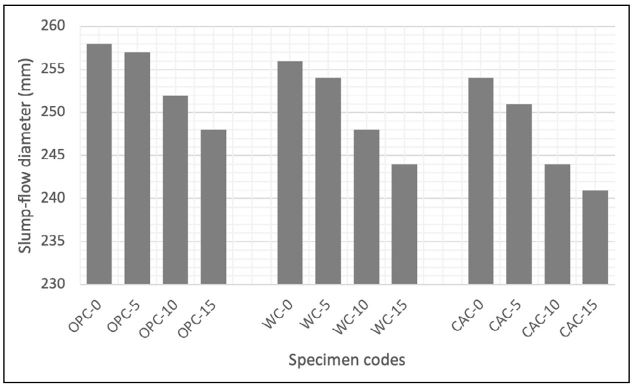

Figure 2. Slump-flow diameters of mixtures.

for OPC-0, OPC-5, OPC-10, and OPC-15, respectively. The slump-flow diameters of the WC-based mixes ranged from 24.4 to 25.6 cm. The lower flow diameter of WC-based mixtures compared to OPC can be attributed to the higher specific surface area of WC (4600 cm^2/g) compared to OPC (3450 cm^2/g). The most significant reduction in flow diameter was obtained in CAC-based mixtures. While the flow diameter of the CAC-0 series was 254 mm, this value was measured as 241 mm in the CAC-15 series.

4.2.Properties of Mortar Specimens Before Freeze-Thaw Cycles

4.2.1. Compressive Strength

The 28-day compressive strength results of SCM specimens before freezing and thawing are given in Figure 3.

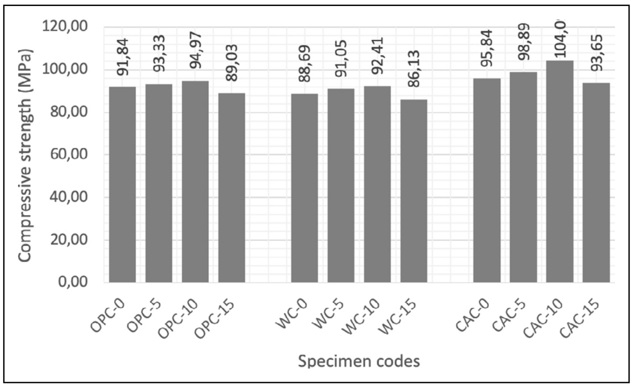

Figure 3. Compressive strength results of mixtures.

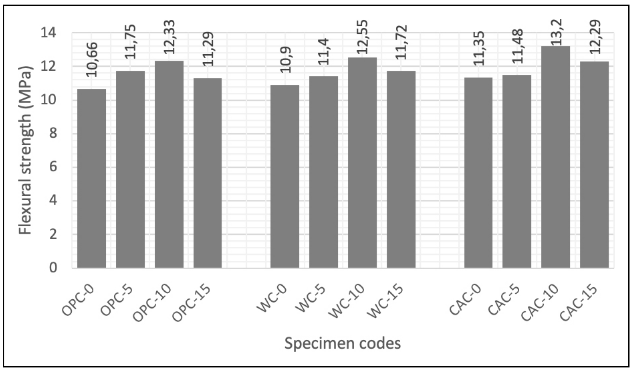

Figure 4. Flexural strength results of mixtures.

Adding WMP up to 10% in all cement series increased compressive strength. This result can be attributed to fine WMP enhancing the properties of the transition zone surrounding the aggregate through its pore-filling effect [10]. As a result of the replacement of cement with 5% WMP at 28-day curing ages, an increase of 1.62%, 2.66%, and 3.18%, respectively, in the compressive strength of the OPC, WC, and CAC series was observed. When replacing 10% WMP with cement, it was determined that the compressive strength of OPC, WC, and CAC series increased by 3.41%, 4.19%, and 8.60%, respectively. Ashish [5] found that adding 10% marble powder instead of cement increased the compressive strength by 8.44% compared to the control specimen. Aliabdo et al. [10] detected that using 5% and 10% WMP caused an increase in compressive strength of 1% and 12%, respectively. It is observed that the strength of the mortar slightly decreased at the 15% level of WMP used as a cement substitute. This is due to reduced cementing materials such as C_3S, C_2S, and C_3A. While 15% WMP substitution reduced the strength by 3.06% in the OPC mixtures, it decreased it by 2.88% and 2.29%, respectively, in the WC and CAC mixtures.

Ergün [11] observed that substituting 5% and 7.5% of cement with WMP increases the compressive strength and decreases the strength by 15%. The optimum WMP ratio was determined as 10% for all cement types. In their research, Vardhan et al. [32] reported that the substitution of 10% WMP is optimum for cement in terms of workability and compressive strength.

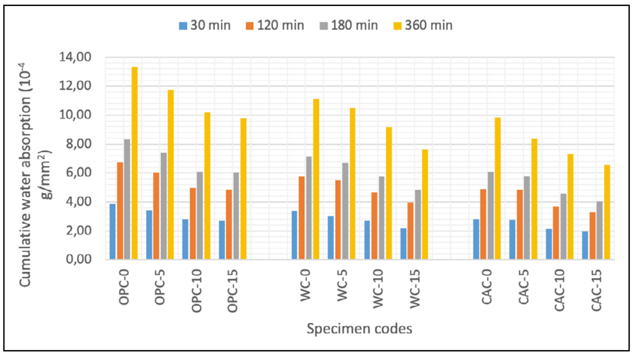

Figure 5. Cumulative capillary water absorption results of mixtures.

As a result of examining the compressive strength in cement types, the highest compressive strength value in all WMP replacement ratios was obtained in the mixtures using CAC. The highest compressive strength value of 104.09 MPa was found in CAC using 10% WMP in all mixes. Although WMP is not pozzolanic, it is not entirely inert because it can react with the alumina phases of cement [9]. If there is an excess of C_3A in the cement, carbo aluminate will be produced from the reaction between $CaCO_3$ and C_3A in WMP [33, 34]. This reaction, which increases compressive strength, increases with the C_3A content in the cement (OPC and WC). In the series without WMP, the highest compressive strength value was obtained from the CAC-0 series, while this value was 8.06% and 4.35% higher compared to the WC-0 and OPC-0 series. In the series using 5%, 10%, and 15% WMP, the compressive strength of CAC-based mixtures was 5.96%, 9.60%, and 5.19% higher, respectively, than OPC. Idrees et al. [35] investigated the properties of CAC and OPC at different curing temperatures using various mineral additives. As a result of the study, they observed that the 28 and 90-day strength values of the CAC-based mixtures were higher than the mixtures with OPC at low curing temperatures (20^{0}C). The high early strength of CAC compared to OPC was attributed to the formation of CAH_{10} and C_2AH_8, which are the dominant hydration products of CAC at low curing temperatures. The compressive strength values of WC-based mixtures using 0%, 5%, 10%, and 15% WMP were approximately 3.43%, 2.44%, 2.70% and 3.26% lower than OPC-based mixtures, respectively. The higher surface area of WP compared to OPC resulted in a decrease in its workability. This may cause small voids in SCMs that self-compact under their weight without requiring additional processing. As a result, this phenomenon may be why the compressive strength of OPC-based mixtures is slightly higher than that of WP-based mixtures.

4.2.2. Flexural Strength

The flexural strength results of the mixtures are given in Figure 4. The highest flexural strength was obtained from the CAC-10 series with 13.20 MPa. Similar to the compressive strength results, flexural strength increased in all cement types up to 10% WMP use. Substitution of 5% and 10% WMP in OPC-based blends increased flexural strength by 10.23% and 15.67% compared to the OPC-0 blend. Ergün

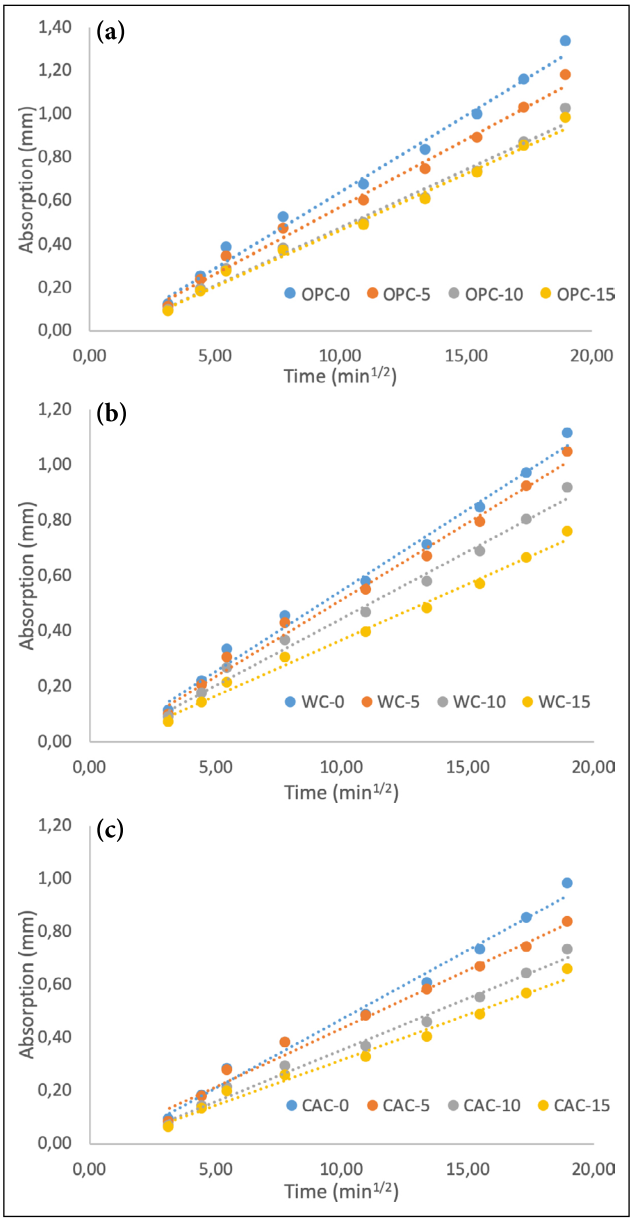

Figure 6. SCM specimens absorption (mm) **(a)** OPC-based mixtures, **(b)** WC-based mixtures, **(c)** CAC-based mixtures.

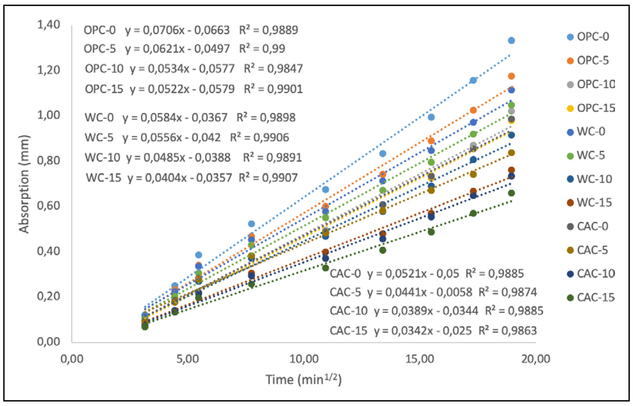

Figure 7. Sorptivity coefficient results of mixtures.

[11] observed that using WMP did not cause a significant change in the relative flexural strength of the mixture samples. As a result of the study, a 5% increase in the 90-day flexural strength of mixtures containing 5% WMP was reported compared to the reference sample. Kumar et al. [36] stated that 5% WMP replacement increased the flexural strength of the mixtures by 3% and 7% in 7 and 28 days, respectively. It was observed that 5% WMP substitution increased the flexural strength of WC and CAC-based mixtures by 4.59% and 1.15% compared to WC-0 and CAC-0 mixtures. 10% WMP substitution improved flexural strength by 15.14% and 16.30% compared to WC and CAC-based reference specimens. This can be attributed to the positive effect of WMP substitution at low rates, as WMP reduces the porosity of mortar samples. In addition, 5% WMP did not significantly affect WC and OPC-based mixtures, while using 10% WMP significantly improved the strength.

Although adding 15% WMP in all cement types decreased the flexural strength, it was higher than the reference specimens. The flexural strength of the OPC-15, WC-15, and CAC-15 series was 5.91%, 7.52%, and 8.28% higher than the control specimens.

4.2.3. Capillary Water Absorption and Sorptivity Coefficients

Capillary water absorption values of the mixtures are presented in Figure 5. Capillary water absorption values decreased with increased WMP ratio in all cement types. This can be attributed to the reduction of porosity in mortar specimens due to the filling effect of WMP use. Topçu [37] reported that adding WMP fills the voids in self-compacting concrete (SCC) and reduces capillary voids due to high workability. Aliabdo et al. [10] reported that the porosity of concrete decreased with the increase of WMP in the case of partial substitution of cement with WMP for different w/b ratios. Increasing the WMP ratio from 0 to 15 in OPC-based mixtures decreased the 6-hour surface water absorption value by 26.7%. Increasing the WMP ratio from 0% to 15% in WC and CAC-based mixtures decreased the capillary water absorption value by 31.65% and 33.30%, respectively. The least capillary water absorption was obtained from CAC-based mixtures among the different cement types. Ashish et al. [38] observed that substituting WMP instead of cement in concrete reduced the water absorption rates of concrete mixtures. Gupta et al. [39] stated that substituting up to 10% WMP reduced water absorption. This result is attributed to the pore-filling effect decreasing the void percentage due to the fineness of the WMP. Khodabakhshian et al. [40] and Zhang et al. [41] determined that the substitution of 5% SF along with 5–20% WMP reduces water absorption due to additional C-S-H gel filling the pores and improving the microstructure. The absorption and time relationship of OPC, WC, and CAC-based mixtures are presented in Figure 6a–c, respectively.

To calculate the sorptivity coefficient, the amount of water adsorbed (mm^3) per the cross-section of the specimen in contact with water (cm^2) (Q/A) was plotted against the square root of time (t), then k was determined from the slope of the linear relationship between Q/A and t. The sorptivity and correlation coefficients of all mixtures are given in Figure 7. As can be seen from Figure 7, the sorptivity coefficient decreased as the WMP ratio increased for all cement types. Due to the small particle size of WMP, the pores at the interfaces between the paste or aggregate and the cement paste are filled with WMP, resulting in smaller capillary pores. The lowest sorptivity coefficient in OPC-based mixtures was obtained from the mixture series using 15% WMP with 0.0522 mm/min$^{1/2}$. This value was 26.06% lower than the OPC-0 series without WMP. Adding 5%, 10%, and 15% WMP in WC-based mixtures decreased the sorptivity coefficient by 4.79%, 16.95% and 30.82%, respectively. The lower sorptivity values of WC-based mixtures compared to OPC-based mixtures can be attributed to the reduction of capillary pores due to the finer particle size of WC compared to OPC. The lowest sorptivity value among all mixes was calculated with 0.0342 mm/min$^{1/2}$ in the CAC-15 series. This value was 34.36% lower compared to the CAC-0 series. In addition, the sorptivity value of the CAC series using 15% WMP was 34.48% and 15.34% lower, respectively, compared to the OPC-15 and WC-15 series. The lower sorptivity coefficient of CAC-based mixtures compared to OPC and WC can be explained by the denser structure of CAC's metastable phases (CAH_{10} and C_2AH_8) compared to the C-S-H phases in OPC. Moffatt [42] reported that CAC samples had a lower chloride diffusion coefficient than OPC samples and attributed this to the denser structure of CAC.

4.3. Properties of Mortar Specimens After Freeze-Thaw Cycles

4.3.1. Residual Compressive Strength

All mixture specimens were subjected to freeze-thaw cycles after a 28-day curing period. The number of cycles was determined as 30, 60, and 90. After the process counts, no significant deterioration occurred in the specimens, which can be attributed to the high amount of binder. Residual compressive strength results of the mixtures after 30, 60, and 90 cycles are presented in Figure 8. The reduction in compressive strength of OPC-based mixtures after 30 cycles varies between 3.1% and 5.3%. While the least strength drop was obtained in the OPC-5 series, the highest decrease was calculated in the OPC-15 series. Similar to the compressive strength results before the freeze-thaw cycles, the residual compressive strengths of the mixtures using 5% and 10% WMP were higher than the OPC-0 series.

After 60 freeze-thaw cycles of the OPC-based mixtures, the residual compressive strengths of the OPC-0, OPC-5, and OPC-10 series were obtained as 86.39 MPa, 87.95 MPa, and 89.26 MPa, respectively. However, despite being high in compressive strength, the relative residual compressive strength (the ratio of residual compressive strength after freeze-thaw to initial compressive strength) was determined as 0.941, 0.942 and 0.940 at 0%, 5%, and 10% WMP change, respectively. A similar situation was observed after 90 cycles. Although the residual compressive strength of the OPC-5 and OPC-10 mixture series was higher than the OPC-0 series, the relative residual compressive strength was determined as 0.893, 0.892, and 0.891 for the OPC-0, OPC-5 and OPC-10 mixtures, respectively. As a result, the addition of 5% and 10% WMP did not cause a significant effect on the strength of the mixtures after 60 and 90 freeze-thaw cycles. This can be attributed to the fact that the C-S-H gel content decreased due to WMP without pozzolanic activity, but the low range (5% and 10%) of WMP compensated for the decrease in strength due to the filling effect. Ince et al. [16] reported that concrete samples containing silica fume and waste marble powder suffered less strength loss after freeze-thaw cycles than the reference sample. Gencel et al. [43] stated that using waste marble powder instead of aggregate in concrete paving blocks increases the freeze-thaw resistance. With an increased WMP ratio of 15%, the freeze-thaw resistance of OPC-based mixtures decreased. After 60 and 90 cycles, the relative residual compressive strength of the OPC-15 series was obtained as 0.930 and 0.864. Increases in the amount (%15) of WMP cause a further reduction of hydration products, creating a loose structure that will increase free water and expansion stress during freeze-thaw cycles, resulting in more strength loss.

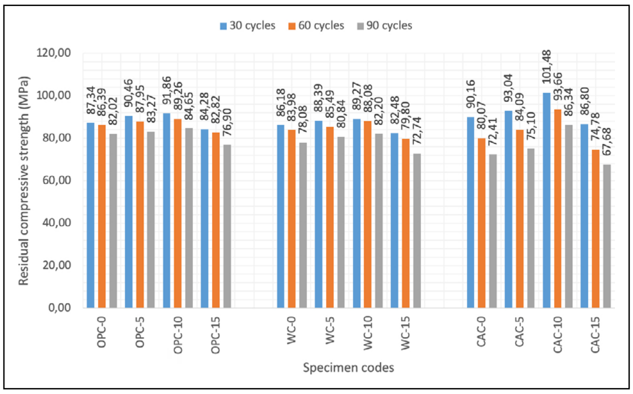

Figure 8. Residual compressive strengths after freeze-thaw.

When the compressive strengths of WC-based mixtures after freeze-thaw cycles are examined in Figure 8, it is observed that the strength loss after 30 cycles varies between 2.8% and 4.2%. When the cycle number increased to 60 in WC-based mixtures, 5.3%, 7.1%, and 4.7% loss occurred in the compressive strength of the mixtures containing 0%, 5%, and 10% WMP, respectively. Similar to the compressive forces before exposure to freeze-thaw, the residual compressive strengths of 5% and 10% WMP were higher compared to the WC-0 series. After 90 freeze-thaw cycles, 0%, 5%, and 10% WMP substitution to WC-based mixes reduced the relative residual compressive strength to 0.880, 0.888, and 0.890, respectively. Similar to OPC-based mixtures, using WMP in low proportions showed a filling effect, making the mortar structure denser and preventing a further decrease in strength despite the decrease in C-S-H structure. In the case of 15% WMP addition, the reduction in strength after 60 and 90 cycles was obtained as 7.3% and 15.5%, respectively. The freezing and thawing resistance of concrete or mortar highly depends on the amount of hydration products and pores. Due to the absence of significant differences in the chemical composition of OPC and WC, the strength drops after cycles are also significantly similar.

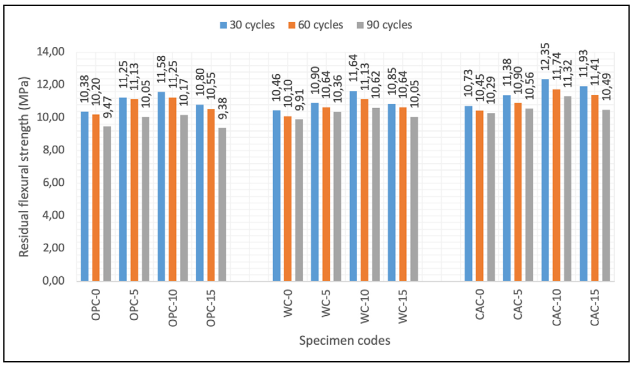

Figure 9. Residual flexural strengths after freeze-thaw.

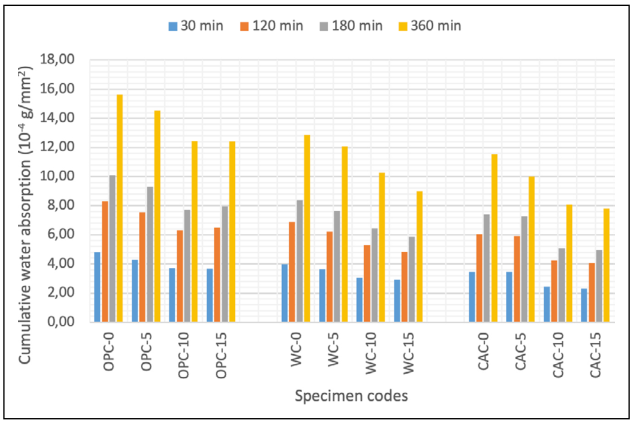

Figure 10. Cumulative capillary water absorption results after 30 freeze-thaw cycles.

When the results of CAC-based mixtures are examined, the reductions in strength after 30 cycles range from 2.5% to 7.3%. The least strength loss was calculated from the CAC-10 series, and the maximum strength reduction was calculated from the CAC-15 series. Significant strength losses occurred in CAC-based mixtures when the number of cycles increased to 60. After 60 freeze-thaw cycles, strength loss happened in the CAC-0, CAC-5, CAC-10, and CAC-15 series at 16.5%, 15%, 10% and 20.1%, respectively. Although there was a decrease in strength loss with an increase in WMP ratio to 10%, the addition of 15% WMP increased the strength loss. After 90 freeze-thaw cycles, the compressive strength loss in CAC-based mixtures using 0%, 5%, 10%, and 15% WMP was obtained as 24.4%, 24.1%, 17.1%, and 27.7%, respectively. Although the compressive strength losses are higher than other cement types, the highest compressive strength was determined in the CAC-10 series in all cycle numbers.

The lower freeze-thaw resistance of CAC-based mixtures compared to other cement types can be explained by the transformation of the metastable phases (CAH_{10} and C_2AH_8), which are the hydration products of CAC, into stable C_3AH_6. With this phase transformation, the porosity of the concrete increases, and its compressive strength decreases [44]. This conversion reaction accelerates at high temperatures and moisture content [45]. As a result, moisture changes in the specimens during the freeze-thaw cycles may cause a decrease in strength by accelerating phase transformations.

4.3.2. Flexural Strength

The flexural strength results of the mixture specimens after freezing and thawing are given in Figure 9. After 30 cycles, the flexural strength of the OPC-based mixtures decreased between 2.6% and 6.1%. When the number of cycles increased to 60, a decrease in bending strength of 4.3%, 5.3%, 8.8%, and 6.6% were detected in the OPC-0, OPC-5, OPC-10, and OPC-15 series, respectively. Relative residual flexural strength values of the mixtures using 0%, 5%, 10%, and 15% WMP after 90 cycles were determined as 0.888, 0.855, 0.825, and 0.831, respectively. Although the strength losses increased with the addition of WMP, the residual flexural strengths were higher than the OPC-0 series without WMP. The highest residual flexural strength of OPC-based mixtures in all cycles was obtained in the OPC-10 series.

Flexural strength loss in WC-0, WC-5, WC-10, and WC-15 mixture series after 30 cycles in WC-based mixtures was determined as 4%, 4.4%, 7.3% and 7.4%, respectively. After 30 cycles, the highest flexural strength was obtained from the WC-10 series. The flexural strength reduction after 60 cycles was determined as 6.7% to 11.3% in WC-based mixtures. After 90 cycles, the strength drops became more pronounced. The relative residual flexural strengths of the WC-0, WC-5, WC-10, and WC-15 mixture series were obtained as 0.909, 0.909, 0.846, and 0.858, respectively. After all cycles, the residual flexural strengths of WMP-added mixes were higher than those without WMP.

As seen in Figure 8, the residual compressive strengths of the WMP-added CAC-based mixtures were higher than the non-WMP mixture after freeze-thaw cycles. Similar to OPC and WC-based mixes, the highest residual flexural strength after all cycles was determined in the CAC-10 series. Using 0.5%, 10%, and 15% WMP in CAC-based mixtures after 30 cycles decreased compressive strength of 5.5%, 0.9%, 6.4%, and 2.9%, respectively. The lowest decrease in strength after 60 cycles was obtained in the CAC-5 series with 5.1%. Strength losses in the CAC-10 and CAC-15 series were 11.1% and 7.2%, respectively. After 90 cycles, the relative residual flexural strengths of the CAC-0, CAC-5, CAC-10, and CAC-15 series were determined as 0.907, 0.920, 0.858, and 0.854. Although using WMP in all three cement types increased overall freeze-thaw flexural strength reductions, the residual flexural strengths were still higher than in non-WMP mixtures. This can be attributed to improving the flexural strength of WMP before the freeze-thaw cycles of the mixes using WMP. In all cement types, the highest residual flexural strength after 30, 60, and 90 cycles was observed in the series with 10% WMP replacement.

4.3.3. Capillary Water Absorption and Sorptivity Coefficient

The capillary water absorption and sorptivity values of all cement types after 30 freeze-thaw cycles are given in Figures 10 and 11. After the freeze-thaw cycle, capillary water absorption values increased in all mixtures. As the WMP content in the mixtures increased, a decrease was observed in the capillary water absorption values. After 30 cycles, the 6-hour water absorption value of the OPC-0, OPC-5, OPC-10, and OPC-15 series increased by approximately 17.42%, 23.47%,

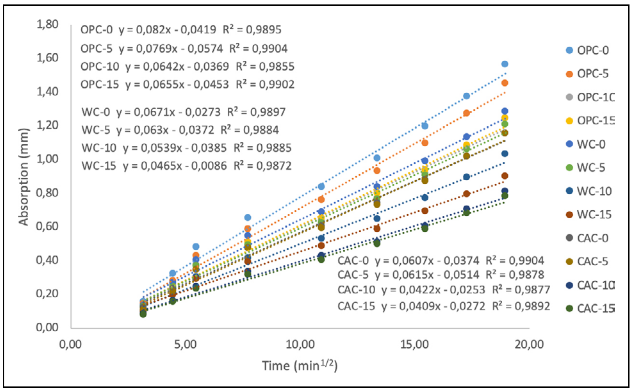

Figure 11. Sorptivity coefficient results after 30 freeze-thaw cycles.

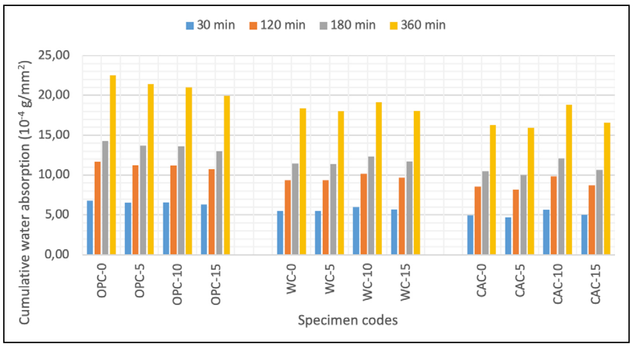

Figure 12. Cumulative capillary water absorption results after 60 freeze-thaw cycles.

21.96%, and 27.05%, respectively, compared to before exposure to freeze-thaw. This was obtained as 15.47%, 15.27%, 12.23%, and 18.42% for the WC-0, WC-5, WC-10, and WC-15 series, respectively. The capillary water absorption of CAC-10, which has the highest strength after 30 freeze-thaws, was obtained as the lowest value at 10.38%. It was determined that the capillary water absorption values of 0%, 5%, and 15% WMP substituted mixtures before freezing and thawing increased by 17.07%, 19.62%, and 18.90%, respectively.

As the WMP ratio in the mixtures increased, the sorptivity values decreased. This can be attributed to the filling effect of WMP. The lowest sorptivity value after 30 freeze-thaw was obtained from the CAC-15 series with 0.0409 mm/min$^{1/2}$. The sorptivity values of OPC-based mixtures vary between 0.0655 and 0.082 mm/min$^{1/2}$. The sorptivity values of the WC-based mixtures were lower compared to the OPC-based mixtures. This can be attributed to WC's smaller average grain size than OPC. The lowest sorptivity values in all cement types occurred in CAC-based mixtures. In CAC-based mixtures, the sorptivity value decreased from 0.0607 mm/min$^{1/2}$ to 0.0409 mm/min$^{1/2}$ as the WMP ratio increased.

Capillary water absorption and sorptivity values after 60 freeze-thaw cycles are presented in Figures 12 and 13. After 60 freeze-thaw cycles, the highest capillary water absorption value in OPC-based mixtures was obtained from the OPC-0 series with 22.52x10^{-4} g/mm^2. Capillary water absorption values decreased as the WMP ratio increased in OPC-based mixes. As the WMP ratio increased in WC and CAC-based mixtures, the capillary water absorption values, except for the CAC-10 series, approached. This shows that the degradation of hydration products is more important than the effect of WMP as the number of cycles increases.

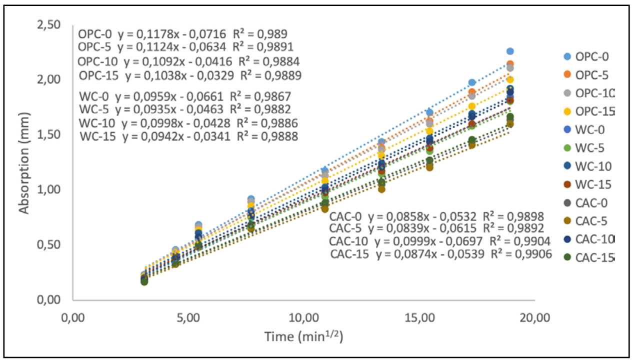

Figure 13. Sorptivity coefficient results after 60 freeze-thaw cycles.

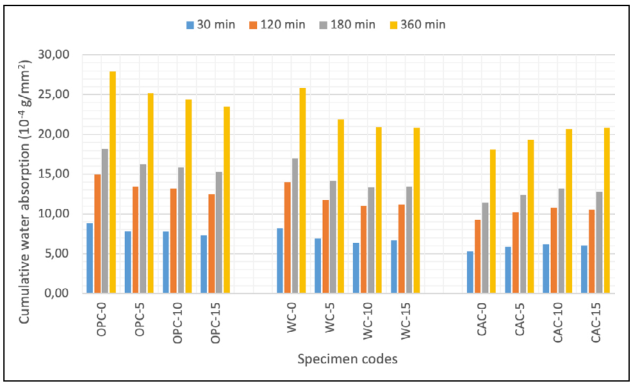

Figure 14. Cumulative capillary water absorption results after 60 freeze-thaw cycles.

After 60 cycles, the sorptivity of the OPC-based mixtures ranged from 0.1038 mm/min$^{1/2}$ to 0.1178 mm/min$^{1/2}$. Similar to capillary water absorption values, the increase in WMP decreased the sorptivity in OPC-based mixtures. After 60 cycles, the OPC-0, OPC-5, OPC-10, and OPC-15 series showed an increase in sorptivity of approximately 66%, 80%, 104%, and 98%, respectively, compared to the before freeze-thaw cycles. The lowest sorptivity value of 0.0935 mm/min$^{1/2}$ in WC-based mixtures was determined in the WC-5 series. After 60 cycles, the sorptivity values of the WC-based mixtures increased in the range of approximately 64% to 133% compared to the initial sorptivity values. The lowest sorptivity values were obtained from CAC-based mixtures. However, the increase ratio compared to the initial sorptivity values is higher than other cement types. The increase in porosity can explain this situation as a result of the transformation of metastable phases in CAC into stable phases. In the CAC-0, CAC-5, CAC-10 and CAC-15 series, these values were 64%, 90%, 156% and 155%, respectively. This may be the reason for significant reductions in compressive strength compared to other cement types.

Capillary water absorption and sorptivity values after 90 freeze-thaw cycles are presented in Figures 14 and 15. Capillary water absorption values decreased as the WMP

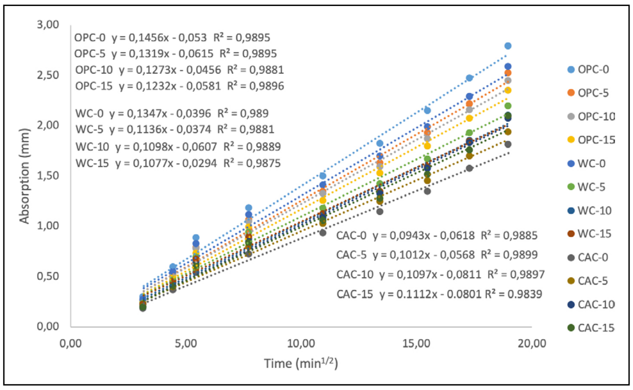

Figure 15. Sorptivity coefficient results after 90 freeze-thaw cycles.

ratio increased in OPC and WC-based mixtures. However, the opposite situation is seen in CAC-based mixtures, and capillary water absorption increased with increased WMP.

When the sorptivity values after 90 cycles were examined, the lowest sorptivity in OPC-based mixtures was obtained in the OPC-15 series. The sorptivity of OPC-based mixtures ranges from 0.1232 mm/min$^{1/2}$ to 0.1456 mm/min$^{1/2}$. In addition, 106%, 112%, 138%, and 136% increases were detected in the OPC-0, OPC-5, OPC-10, and OPC-15 series, respectively, according to the sorptivity before exposure to freeze-thaw cycles. In WC-based mixtures, the sorptivity values decreased as WMP increased. Sorptivity values vary between 0.1077 mm/min$^{1/2}$ and 0.1347 mm/min$^{1/2}$. Contrary to OPC and WC, the increase in WMP ratio increased the sorptivity value in CAC-based mixtures. In contrast to OPC and WC, it was observed that CAC and WMP reacted to increase the strength before exposure to freeze-thaw. The sorptivity values of the CAC-0, CAC-5, CAC-10, and CAC-15 series were determined as 0.0943, 0.1012, 0.1097, and 0.1112 mm/min$^{1/2}$, respectively. These values were approximately 81%, 129%, 182%, and 222% higher than the baseline values. When this situation is examined, it can be thought that the capillary void ratio increases significantly compared to other cement types. As a result, it causes significant decreases in compressive strength.

5. CONCLUSIONS

- While adding 5% WMP did not affect the slump-flow diameter much, the flow diameter decreased as the use of WMP increased. However, the slump-flow diameter of all mixtures was in the range of 24 to 26 cm. The greatest loss of workability was observed in CAC-based blends.
- The highest compressive strength values before freeze-thaw cycles were obtained from CAC-based mixtures. The compressive strengths of CAC-based mixtures with 5% and 10% WMP replacement were obtained as 98.89 MPa and 104.09 MPa, respectively. The compressive strengths of the OPC and WC-based mixtures were not significantly different.
- The highest flexural strength was obtained from the CAC-10 series with 13.20 MPa. The flexural strength of the OPC-15, WC-15, and CAC-15 series was 5.91%, 7.52%, and 8.28% higher than the control specimens.
- The most appropriate WMP ratio was 10% in mechanical properties before the freeze-thaw cycles. Decreases in the strength of CAC-based mixtures after 30 cycles vary between 2.5% and 7.3%. This was calculated between 3.1%–5.3% and 2.8%–4.2% in OPC and WC-based mixtures, respectively.
- The mixtures with the lowest sorptivity values before the freeze-thaw cycles were CAC-based, and this situation was similar to the strength results.
- Significant strength reductions occurred in CAC-based mixtures, especially at 60 and 90 cycle numbers, in mixtures exposed to freeze-thaw.

ETHICS

There are no ethical issues with the publication of this manuscript.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

FINANCIAL DISCLOSURE

The authors declared that this study has received no financial support.

PEER-REVIEW

Externally peer-reviewed.

REFERENCES

[1] Zareei, S. A., Ameri, F., Bahrami, N., Shoaei, P., Moosaei, H. R., & Salemi, N. (2019). Performance of sustainable high-strength concrete with basic oxygen steel-making (BOS) slag and nano-silica. *J Build Eng*, 25, 100791. [CrossRef]

[2] Shaikh, F. U. A. (2016). Mechanical and durability properties of fly ash geopolymer concrete containing recycled coarse aggregates. *Int J Sustain Built Environ*, 5(2), 277–287. [CrossRef]

[3] Çelik, Z. (2023). Investigation of the use of ground raw vermiculite as a supplementary cement material in self-compacting mortars: Comparison with class C fly ash. *J Build Eng*, 65, 105745. [CrossRef]

[4] Huntzinger, D. N., & Eatmon, T. D. (2009). A life-cycle assessment of Portland cement manufacturing: Comparing the traditional process with alternative technologies. *J Clean Prod*, 17(7), 668–675. [CrossRef]

[5] Ashish, D. K. (2018). Feasibility of waste marble powder in concrete as partial substitution of cement and sand amalgam for sustainable growth. *J Build Eng*, 15, 236–242. [CrossRef]

[6] Bostanci, S. C. (2020). Use of waste marble dust and recycled glass for sustainable concrete production. *J Clean Prod*, 251, 119785. [CrossRef]

[7] Vardhan, K., Siddique, R., & Goyal, S. (2019). Strength, permeation, and micro-structural characteristics of concrete incorporating waste marble. *Constr Build Mater*, 203, 45–55. [CrossRef]

[8] Munir, M. J., Kazmi, S. M. S., & Wu, Y. F. (2017). Efficiency of waste marble powder in controlling alkali–silica reaction of concrete: A sustainable approach. *Constr Build Mater*, 154, 590–599. [CrossRef]

[9] Uysal, M., & Yilmaz, K. (2011). Effect of mineral admixtures on properties of self-compacting concrete. *Cem Concr Compos*, 33(7), 771–776. [CrossRef]

[10] Aliabdo, A. A., Abd Elmoaty, M., & Auda, E. M. (2014). Re-use of waste marble dust in the production of cement and concrete. *Constr Build Mater*, 50, 28–41. [CrossRef]

[11] Ergün, A. (2011). Effects of the usage of diatomite and waste marble powder as partial replacement of cement on the mechanical properties of concrete. *Constr Build Mater*, 25(2), 806–812. [CrossRef]

[12] Rodrigues, R., De Brito, J., & Sardinha, M. (2015). Mechanical properties of structural concrete containing very fine aggregates from marble cutting sludge. *Constr Build Mater*, 77, 349–356. [CrossRef]

[13] Zhao, J., Cai, G., Gao, D., & Zhao, S. (2014). Influences of freeze–thaw cycle and curing time on chloride ion penetration resistance of sulphoaluminate cement concrete. *Constr Build Mater*, 53, 305–311. [CrossRef]

[14] Wang, R., Zhang, Q., & Li, Y. (2022). Deterioration of concrete under the coupling effects of freeze–thaw cycles and other actions: A review. *Constr Build Mater*, 319, 126045. [CrossRef]

[15] Keleştemur, O., Yildiz, S., Gökçer, B., & Arici, E. (2014). Statistical analysis for freeze–thaw resistance of cement mortars containing marble dust and glass fiber. *Materials & Design*, 60, 548–555. [CrossRef]

[16] Ince, C., Hamza, A., Derogar, S., & Ball, R. J. (2020). Utilisation of waste marble dust for improved durability and cost efficiency of pozzolanic concrete. *J Clean Prod*, 270, 122213. [CrossRef]

[17] Karakurt, C., & Dumangöz, M. (2022). Rheological and Durability Properties of Self-Compacting Concrete Produced Using Marble Dust and Blast Furnace Slag. *Materials*, 15(5), 1795. [CrossRef]

[18] Gencel, O., Benli, A., Bayraktar, O. Y., Kaplan, G., Sutcu, M., & Elabade, W. A. T. (2021). 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, 123374. [CrossRef]

[19] Burris, L., Kurtis, K., & Morton, T. (2015). *Novel alternative cementitious materials for development of the next generation of sustainable transportation infrastructure* [Tech Brief]. United States. Federal Highway Administration.

[20] Scrivener, K. L., Cabiron, J. L., & Letourneux, R. (1999). High-performance concretes from calcium aluminate cements. *Cem Concr Res*, 29(8), 1215–1223. [CrossRef]

[21] Li, G., Zhang, A., Song, Z., Shi, C., Wang, Y., & Zhang, J. (2017). Study on the resistance to seawater corrosion of the cementitious systems containing ordinary Portland cement or/and calcium aluminate cement. *Constr Build Mater*, 157, 852–859. [CrossRef]

[22] Son, H. M., Park, S., Kim, H. Y., Seo, J. H., & Lee, H. K. (2019). Effect of CaSO4 on hydration and phase conversion of calcium aluminate cement. *Constr Build Mater*, 224, 40–47. [CrossRef]

[23] Eren, F., Keskinateş, M., Felekoğlu, B., & Felekoğlu, K. T. (2023). Effects of mineral additive substitution on the fresh state and time-dependent hardened state properties of calcium alumina cement mortars [Article in Turkish]. *Turkish J Civ Eng*, 34(3), 139–162. [CrossRef]

[24] TS EN 1097–6 (2013). *Tests for mechanical and physical properties of aggregates - part 6: determination of particle density and water absorption*. Turkish Standards. Ankara, Turkiye.

[25] EFNARC (2002). *Specifications and guidelines for self-compacting concrete*. EFNARC.

[26] ASTM C109 (2007). *Standard test method for compressive strength of hydraulic cement mortars (Using 2-in. or [50-mm] Cube specimens)*. Annual Book of ASTM Standards.

[27] ASTM C348-02. *Standard test method for flexural strength of hydraulic-cement mortars*. ASTM C348. Annual Book of ASTM Standards.

[28] ASTM C1585-04. *Standard test method for measurement of rate of absorption of water by hydraulic-cement concretes*.

[29] ASTM C 666 (2003). *Standard test method for resistance of concrete to rapid freezing and thawing*.

[30] Rashwan, M. A., Al-Basiony, T. M., Mashaly, A. O., & Khalil, M. M. (2020). Behaviour of fresh and hardened concrete incorporating marble and granite sludge as cement replacement. *J Build Eng*, 32, 101697. [CrossRef]

[31] Li, L. G., Huang, Z. H., Tan, Y. P., Kwan, A. K. H., & Liu, F. (2018). Use of marble dust as paste replacement for recycling waste and improving durability and dimensional stability of mortar. *Constr Build Mater*, 166, 423–432. [CrossRef]

[32] Vardhan, K., Goyal, S., Siddique, R., & Singh, M. (2015). Mechanical properties and microstructural analysis of cement mortar incorporating marble powder as partial replacement of cement. *Constr Build Mater*, 96, 615–621. [CrossRef]

[33] Bonavetti, V. L., Rahhal, V. F., & Irassar, E. F. (2001). Studies on the carboaluminate formation in limestone filler-blended cements. *Cem Concr Res*, 31(6), 853–859. [CrossRef]

[34] Péra, J., Husson, S., & Guilhot, B. (1999). Influence of finely ground limestone on cement hydration. *Cem Concr Compos*, 21(2), 99–105. [CrossRef]

[35] Idrees, M., Ekincioglu, O., & Sonyal, M. S. (2021). Hydration behavior of calcium aluminate cement mortars with mineral admixtures at different curing temperatures. *Constr Build Mater*, 285, 122839. [CrossRef]

[36] Kumar, V., Singla, S., & Garg, R. (2021). Strength and microstructure correlation of binary cement blends in presence of waste marble powder. *Mater Today Proceedings*, 43, 857–862. [CrossRef]

[37] Topcu, I. B., Bilir, T., & Uygunoğlu, T. (2009). Effect of waste marble dust content as filler on properties of self-compacting concrete. *Constr Build Mater*, 23(5), 1947–1953. [CrossRef]

[38] Ashish, D. K., Verma, S. K., Kumar, R., & Sharma, N. (2016). Properties of concrete incorporating sand and cement with waste marble powder. *Adv Concr Constr*, 4(2), 145. [CrossRef]

[39] Gupta, R., Choudhary, R., Jain, A., Yadav, R., & Nagar, R. (2021). Performance assessment of high strength concrete comprising marble cutting waste and fly ash. *Mater Today Proceedings*, 42, 572–577. [CrossRef]

[40] Khodabakhshian, A., De Brito, J., Ghalehnovi, M., & Shamsabadi, E. A. (2018). Mechanical, environmental and economic performance of structural concrete containing silica fume and marble industry waste powder. *Constr Build Mater*, 169, 237–251. [CrossRef]

[41] Zhang, S., Cao, K., Wang, C., Wang, X., Wang, J., & Sun, B. (2020). Effect of silica fume and waste marble powder on the mechanical and durability properties of cellular concrete. *Constr Build Mater*, 241, 117980. [CrossRef]

[42] Moffatt, E. (2016). *Durability of rapid-set (ettringite-based) binders* [Dissertation]. University of New Brunswick.

[43] Gencel, O., Ozel, C., Koksal, F., Erdogmus, E., Martínez-Barrera, G., & Brostow, W. (2012). Properties of concrete paving blocks made with waste marble. *J Clean Prod*, 21(1), 62–70. [CrossRef]

[44] Ahmed, A. A., Shakouri, M., Trejo, D., & Vaddey, N. P. (2022). Effect of curing temperature and water-to-cement ratio on corrosion of steel in calcium aluminate cement concrete. *Constr Build Mater*, 350, 128875. [CrossRef]

[45] Matusinović, T., Šipušić, J., & Vrbos, N. (2003). Porosity–strength relation in calcium aluminate cement pastes. *Cem Concr Res*, 33(11), 1801–1806. [CrossRef]