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Abstract: The demand for cement is increasing every day worldwide. To meet this demand, natural resources are rapidly being depleted. The excessive consumption of natural resources encourages researchers to conduct studies on the use of waste materials instead of cement. Marble waste is one of the major natural wastes abundantly generated worldwide. It has been evaluated that there is a gap in the literature regarding a study comparing the effects of different cooling regimes on cementitious composites with a marble powder (MP) replacement that has been exposed to high temperatures. In this study, waste marble powder (MP) was used as a replacement for cement at percentages of 5%, 10%, 15%, 20%, and 25% by mass. The water-to-binder ratio was kept constant at 0.5 for all mixture groups. Subsequently, the prepared cementitious composites were exposed to high temperatures (300 °C, 600 °C, and 800 °C) and subjected to air- and water-cooling regimes. Within the scope of this study, unit weight ( $U_w$ ), ultrasonic pulse velocity (UPV), flexural strength ( $f_{fs}$ ), compressive strength  $(f_{cs})$ , and mass loss tests were conducted. Additionally, a microstructure analysis was carried out using scanning electron microscopy (SEM) to examine the effect of MP replacement and the cooling regime. When examining the results of the samples tested in the laboratory, it was observed that the mortar with 5% MP replacement exhibited better mechanical properties compared with the others. In general, it can be said that the mechanical properties of samples cooled in air after exposure to high temperatures were better than those of samples cooled in water. As a result of this study, it was determined that MP replacement could positively contribute to the resistance of cementitious composites to high temperatures. Additionally, the use of a significant amount of waste MP can lead to savings in cement usage and significant reductions in CO<sub>2</sub> emissions.

**Keywords:** different cooling regimes; elevated temperatures; microstructure of cementitious composites; marble powder; mechanical properties of cement mortar

# 1. Introduction

The construction sector stands out as one of the industries benefiting the most from technological advancements. Due to technological developments in the sector, the production of ready-mix concrete is increasing day by day. However, this situation brings about specific issues. Some of these issues include increased raw material consumption in concrete production, depletion of nonrenewable resources, and increased CO<sub>2</sub> emissions due to higher cement consumption [1,2]. The depletion of raw material resources and the escalation of environmental pollution have prompted researchers to conduct various studies for the sustainability of the construction industry. In some studies, the usability of industrial waste has been investigated in cementitious composites, geopolymers, asphalt coatings, and road base and subbase layers, either as aggregates or binders [3–9].

Significant amounts of waste are generated during the production of marble, which has been one of the most widely used building materials since ancient times. Approximately 30% of marble is wasted during processing, leading to substantial waste generation [10]. Waste marble in the form of dust causes significant environmental damages such as air



Citation: Bayer, I.R.; Sevim, O.; Demir, I. Effects of High Temperature and Cooling Regimes on Properties of Marble Powder-Based Cementitious Composites. *Buildings* 2023, *13*, 2527. https://doi.org/10.3390/ buildings13102527

Academic Editor: Hamzeh Hajiloo

Received: 18 September 2023 Revised: 3 October 2023 Accepted: 4 October 2023 Published: 6 October 2023



**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). pollution, clogging of sewage systems, obstruction of aquifer soil pores, disruption of vegetation cover, and threats to human health [11,12]. These environmental impacts have prompted researchers to investigate the utilization of waste marble powder in cementitious composites. In some studies, it has been reported that the incorporation of marble powder yields positive results in the properties of cementitious composites [13–15].

Aliabdo et al. [10] investigated using marble powder (MP) in cement and concrete production. MP was incorporated into mixtures in two different ways, as a replacement for sand and cement, up to a proportion of 15%. Overall, they reported that the inclusion of MP in concrete mixtures as a cement or sand replacement gradually improved concrete's physical and mechanical properties, particularly at lower water/cement ratios. Vardhan et al. [16] replaced cement with MP at proportions of 10%, 20%, 30%, 40%, and 50%. The study examined MP-substituted cement pastes' setting behavior, soundness, flowability, strength development, and microstructure properties. It was determined that MP can be used up to a substitution rate of 10% without adversely affecting mixture properties. Singh et al. [17] investigated the influence of dried waste marble slurry, used as a substitute for cement, on concrete's fresh and hardened properties. They found that using dried waste marble slurry with up to a 15% substitution rate led to enhanced mechanical properties for concrete.

Additionally, they reported that marble slurry improved the compactness and uniformity of the concrete structure, acting as a filler and reducing drying shrinkage. Siddique et al. [18] conducted a study on the supplementary cementitious behavior of raw marble slurry and calcined marble slurry. The study explored compressive strength, microstructure analysis, and physicomechanical properties. Mixtures prepared with calcined marble slurry exhibited higher compressive strength than those prepared with raw marble slurry. Substituting up to 10% of calcined marble slurry led to a 7% increase in 28-day compressive strength. Additionally, a 10% substitution rate of calcined marble slurry reduced porosity by about 7.5%.

Structures are exposed to various external factors such as wind, earthquakes, and fires throughout their service life. Fire is one of the most significant external factors threatening structures' safety [19]. Structures can be directly or indirectly affected by high temperatures. The design stage should involve considering the resistance characteristics of structures to fire. The construction materials used in a structure are crucial in its fire resistance. Concrete is the most commonly preferred building material in construction. Therefore, understanding the performance of concrete during a fire is of utmost importance. When subjected to high temperatures, concrete undergoes various physical and chemical changes such as color alteration, strength loss, cracking, and fragmentation. This situation has led researchers to investigate the effects of aggregate type, cement type and dosage, fiber materials, supplementary cementitious materials (SCMs), and cooling regimes on cementitious composites exposed to high temperatures [20–24].

The use of supplementary cementitious materials (SCMs) in concrete or mortar is becoming more widespread day by day [25–28]. These materials contribute to saving cement usage and help reduce environmental pollution. SCMs provide positive contributions to the physical and mechanical properties of cementitious composites. When SCM-incorporated mortars are exposed to high temperatures, they can exhibit different characteristics than those without SCM. Yamanel et al. [29] replaced MP with cement at proportions of 5%, 10%, 15%, and 20% and investigated the effect of high temperatures (300 °C, 600 °C, and 900 °C) on cement mortars. The study concluded that substituting MP for cement positively contributes to the workability and resistance against high temperatures of fresh mortar. Jiang et al. [30] replaced waste glass powder with cement at proportions of 10%, 20%, and 30%. The mechanical properties after exposure to the ambient temperature and high temperatures (800 °C, 1000 °C, and 1200 °C), as well as the physical properties of fresh cement pastes, were examined. The results showed that waste glass powder substitution increased workability and extended the setting time.

Marble powder (MP) is known to cause significant environmental pollution. Therefore, it is believed that efforts to manage MP should be intensified. One of the waste disposal methods is to use it as a substitute for cement in mortar. Despite numerous studies in the literature on this topic, there is a limited amount of research focusing on the effects of different cooling regimes on the mechanical and microstructural properties of cement mortars containing MP and exposed to high temperatures. In addition, as far as the authors know, no article compares MP-added mortars exposed to high temperatures and cooled in air and water, which motivated this study. Finally, it is thought that more research should be conducted on the disposal of this waste material (MP), which is released in very high amounts. This study is aimed at investigating the impact of various cooling regimes on MP-substituted mortars. For this reason, cement mortars were prepared with substitutions of 5%, 10%, 15%, 20%, and 25% MP for cement. These mortar mixtures were cured in water for 28 days and then subjected to high temperatures. The cement mortars were exposed to temperatures of 300 °C, 600 °C, and 800 °C in a high-temperature furnace, followed by cooling in air and water. After the cooling process, the unit weight  $(U_w)$ , ultrasonic pulse velocity (UPV), flexural strength ( $f_{fs}$ ), compressive strength ( $f_{cs}$ ), and mass losses of the samples were determined.

Additionally, the samples were tested under laboratory conditions (20  $^{\circ}$ C), and the effects of high temperature on the mortar samples were compared and evaluated. Lastly, microstructural (SEM) analyses were conducted on reference (0% MP) and 10% MP-substituted mortar samples tested under laboratory conditions (20  $^{\circ}$ C) and after exposure to 600  $^{\circ}$ C. By examining the effects of different cooling regimes on MP-substituted mortar, this study aims to contribute to a better understanding of the behavior of these materials under high-temperature conditions.

## 2. Experimental Program

In this section, the materials used in the production of MP-substituted cementitious composites exposed to high temperatures (CEM I 42.5 R, MP, water, and sand), the mixing ratios (5%, 10%, 15%, 20%, and 25% MP replacement of cement by weight), and the applied tests (unit weight, ultrasonic pulse velocity, compressive strength, flexural strength, mass loss, and microstructural analyses) are presented in detail.

#### 2.1. Materials

In cement mortars, CEM I 42.5 R Portland cement (PC) and marble powder (MP) were used as binder materials. Table 1 presents the physical and chemical properties of PC and MP. After sieving, MP was utilized from the portion that remained below the 75-micron (No. 200) sieve. Standard sand defined by EN 196-1 [31] was used as aggregate in the mortar mixtures. Tap water was used as mixing water. CEM I 42.5 R cement is a type of pure cement. This type of cement is preferred in many experimental studies in the literature. In addition, since it was thought that the pure cement type (CEM I 42.5 R) should be chosen to better see pozzolanic materials' effect on cement-based composites, it was deemed appropriate to use this type of cement in this study. As mentioned in the introduction, a considerable amount of MP is released into nature as a waste material. Therefore, with the thought that this material should be disposed of, the authors decided to use it within the scope of this study. When additional cementitious materials such as MP are substituted for cement, they reduce the mortar matrix's  $Ca(OH)_2$  density. When the  $Ca(OH)_2$  density decreases, the strength loss of cementitious composites is reduced, especially at high temperatures. When the temperature reaches around 500  $^{\circ}$ C, Ca(OH)<sub>2</sub>, which constitutes a large part of the material, begins to break down, and significant decreases in strength are observed. For this reason, it was thought that the effect of MP on the properties of cementitious composites exposed to high temperatures should be investigated.

Chemical Composition (%)	РС	МР	
SiO <sub>2</sub>	24.32	10.03	
$Al_2O_3$	3.97	0.72	
Fe <sub>2</sub> O <sub>3</sub>	3.34	1.16	
CaO	59.98	64.52	
MgO	2.01	12.46	
Na <sub>2</sub> O	0.36	0.22	
K <sub>2</sub> O	0.42	0.39	
$SO_3$	4.23	0.25	
Physical Properties			
Specific gravity	3.13	2.61	
Specific surface area $(cm^2/g)$	3395	2720	
Loss on ignition (%)	1.43	9.96	

Table 1. Physical and chemical properties of PC and MP.

#### 2.2. Mixture Proportions

In cement mortars, the amounts of sand, water, and binder were kept constant, and the mortars were prepared in accordance with EN 196-1 [31]. As a substitute for PC, MP was incorporated at different proportions (5%, 10%, 15%, 20%, and 25%). The quantities of materials used in the mortar mixtures and their corresponding codes are presented in Table 2. Fresh mortar mixtures were compacted and poured into molds with dimensions of  $40 \times 40 \times 160$  mm in two layers. After 24 h, the samples were removed from the molds and cured in a water curing tank for 28 days.

Table 2. Mortar mix proportions.

Mixture Code	PC (g)	MP (g)	<b>Replacement of MP (%)</b>	Water (g)	Sand (g)
Ref	450.0	0.0	0	225	1350
MP5	427.5	22.5	5	225	1350
MP10	405.0	45.0	10	225	1350
MP15	382.5	67.5	15	225	1350
MP20	360.0	90.0	20	225	1350
MP25	337.5	112.5	25	225	1350

#### 2.3. Test Procedure

After 28 days of curing, the mortar samples were initially dried in an oven at 105  $\pm$  5  $^{\circ}$ C for 24 h. Subsequently, the mortar samples were exposed to temperatures of 300 °C, 600 °C, and 800 °C. These target temperatures were determined considering the literature's most commonly used high-temperature values [21,32–34]. When studies on the behavior of cement-based composites at high temperatures are examined, the following can be observed: if the temperature rises above 300 °C, decreases in strength begin to be seriously noticed [35], and when it reaches around 500 °C, calcium hydroxide (Ca(OH)<sub>2</sub>), which constitutes a large part of the hydrated products of concrete, begins to break down [36]. It has also been reported in studies that the calcium silicate hydrate (C-S-H) phase begins to decompose at 600 °C and that the C-S-H phase completely decomposes above  $800-900 \,^{\circ}C$  [37]. For this reason, it was thought that cementitious composites should be exposed to temperatures of 300 °C, 600 °C, and 800 °C within the scope of this study. The oven's heating rate was set to 5 °C/min, and once the oven reached the target temperatures, the samples were subjected to these temperatures for 2 h and 30 min. At the end of this period, the samples underwent two different cooling regimes: cooling with air and with water. The oven's heat source was turned off during air cooling, allowing the samples to reach ambient temperature. The temperature during air cooling decreased at an approximate rate of 1.1 °C/min. For water cooling of the samples exposed to high temperatures, water was directly sprayed onto them as they were removed from the oven. Figure 1a,b

illustrate the oven and the heating–cooling regime used in this study. The furnace shown in Figure 1a can reach temperatures of up to 1400 °C. The samples were exposed to high temperatures in this furnace. The graph in Figure 1b shows the heating–cooling regimes of the samples. The furnace reached temperatures from 20 °C to 300, 600, and 800 °C at a constant rate, increasing by 5 °C per minute. The graph demonstrates that it was kept at these temperatures for 2 h and 30 min and then cooled down to 20 °C at a rate of 1.1 °C per minute.



Figure 1. Furnace (a) and heating–cooling regime (b).

The mortar samples' unit weight ( $U_w$ ) measurements were conducted according to the ASTM C642-13 [38] standard. One commonly used nondestructive testing method to determine the quality of mortars is the ultrasonic pulse velocity (UPV) test. The UPV measurements of the mortars were performed with a precision of 0.10 s following the ASTM C 597-16 [39] standard. The concrete quality classes based on UPV values are shown in Table 3 [40,41]. The flexural and compressive strengths of the prismatic mortar samples with dimensions of 40 × 40 × 160 mm were determined per EN 196-1 [31]. In the flexural strength ( $f_{fs}$ ) test, the specimens were tested at a loading rate of 50 ± 10 N/s. After the  $f_{fs}$  test, the samples were split into two parts for the compressive strength ( $f_{cs}$ ) test. The loading rate of the  $f_{cs}$  test apparatus was 2400 ± 200 N/s.

**Table 3.** Quality of concrete as a function of the ultrasonic pulse velocity [40].

UPV (km/s)	>4.5	3.5-4.5	3.0-3.5	2.0-3.0	<2.0
Concrete Quality	Excellent	Good	Doubtful	Poor	Very poor

While measuring the mass losses of the samples, their masses were recorded before exposure to high temperatures and after the high-temperature and cooling regimes. Mass losses were calculated based on these measurements. Before conducting experiments on the samples cooled with water after exposure to high temperatures, these samples were dried for 24 h in an oven at a temperature of  $105 \pm 5$  °C. The microstructure analysis of the mortar samples was conducted using a Zeiss EVO 40XP SEM device on small specimens obtained after the compressive strength test. The workflow diagram of the test procedure is shown in Figure 2. In Figure 2, the mixing, molding, and curing of the materials and the subsequent experimental studies are visually explained with a workflow diagram. In the presented workflow chart, it can be seen that after curing, some of the samples were tested under laboratory conditions, some were tested via cooling in air after being exposed to high temperatures.



Figure 2. Mixture design procedure and experimental flowchart.

## 3. Results and Discussion

## 3.1. Unit Weight

This section presents the mortar samples'  $U_w$  (unit weight) findings. In the first part, the samples tested in the laboratory environment (20 °C) were compared with those aircooled after high-temperature exposure. In the second part, the samples tested in the laboratory environment (20 °C) were compared with the water-cooled samples after high-temperature exposure. In the third part, the samples air-cooled and water-cooled after exposure to high temperatures were compared.

# 3.1.1. The $U_w$ Findings for the Samples Air-Cooled after High-Temperature Exposure

The  $U_w$  (unit weight) values of the samples air-cooled after exposure to high temperatures are presented in Figure 3. When examining the  $U_w$  values of the mortar samples tested in the laboratory environment (20 °C), it can be observed that they vary between 2304 kg/m<sup>3</sup> and 2225 kg/m<sup>3</sup>. It was evident that as the MP substitution ratio increases in the mixtures, the  $U_w$  values decrease. This can be attributed to the lower specific gravity of MP compared with cement [29,42]. Upon analyzing Figure 3, it was observed that the  $U_w$ values decrease as the temperature increases. The  $U_w$  values of the samples subjected to temperatures of 20 °C, 300 °C, 600 °C, and 800 °C varied in the ranges of 2304–2225 kg/m<sup>3</sup>, 2197–2145 kg/m<sup>3</sup>, 2075–2031 kg/m<sup>3</sup>, and 1936–1897 kg/m<sup>3</sup>, respectively.



**Figure 3.** The  $U_w$  values of the samples air-cooled after high-temperature exposure.

The relative residual  $U_w$  percentages of the samples exposed to high temperatures are presented in Figure 4 as a percentage of the  $U_w$  values of the samples tested at laboratory conditions (20 °C). The relative residual  $U_w$  percentages showed a decrease as the temperature values increased. For the mortar samples exposed to temperatures of 300, 600, and 800 °C, the relative residual  $U_w$  percentages were 95.35–96.42%, 90.04–91.27%, and 84.04–85.27%, respectively.



**Figure 4.** Relative residual  $U_w$  of mortars with air cooling.

The decrease in the relative residual  $U_w$  values of the samples after exposure to 300 °C can be attributed to the evaporation of capillary water between the layers of the mortar matrix. As the temperature exceeds 300 °C, the decrease in  $U_w$  values can be attributed to the dissociation of cement components due to the high-temperature effect and the evaporation of bound water in these components. Regardless of the temperature, the relative residual  $U_w$  values increased as the MP substitution ratio increased. This phenomenon can be related to the potential pozzolanic reaction of MP and the lower content of calcium hydroxide (CH) in MP-based mortars at higher temperatures [43].

# 3.1.2. The $U_w$ Findings for Samples Cooled with Water after Exposure to High Temperatures

The  $U_w$  values of samples cooled with water after exposure to high temperatures are presented in Figure 5. The  $U_w$  values of mortar samples cooled with water after high-temperature exposure were lower than those of samples tested in the laboratory environment (20 °C). After exposure to temperatures of 300 °C, 600 °C, and 800 °C, the  $U_w$  values of the samples were in the ranges of 2154–2209 kg/m<sup>3</sup>, 2039–2085 kg/m<sup>3</sup>, and 1922–1960 kg/m<sup>3</sup>, respectively.



**Figure 5.** The  $U_w$  values of samples cooled with water after exposure to high temperatures.

The relative residual  $U_w$  percentages of samples exposed to high temperatures are presented in Figure 6. The relative residual  $U_w$  percentages decreased as the temperature increased. These results exhibited a similar trend to those observed in samples cooled in air. For the mortar samples exposed to temperatures of 300 °C, 600 °C, and 800 °C, the relative residual  $U_w$  percentages were found to be in the ranges of 95.87–96.81%, 90.51–91.65%, and 85.09–86.37%, respectively.



**Figure 6.** Relative residual  $U_w$  of mortars with water cooling.

Similar to the samples cooled in air, it was observed that regardless of the temperature, an increase in the MP substitution rate resulted in higher relative residual  $U_w$  values. The reason for the higher relative residual  $U_w$  values of MP-blended mortars compared with

reference mortars is attributed to the pozzolanic reaction of MP and the fact that MPblended mortars contain a lower amount of Ca(OH)<sub>2</sub> compared with reference mortars [43]. A similar trend in the  $U_w$  values of samples cooled with water was also observed in the study conducted by Alakara et al. [37].

3.1.3. Comparison of  $U_w$  Findings for Samples Cooled in Air and Water after High-Temperature Exposure

The unit weights of samples cooled in air and water after high-temperature exposure are presented in Figure 7. It can be observed that the measured  $U_w$  results for both cooling regimes were lower than those of the samples tested in the laboratory environment (20 °C).



**Figure 7.** The  $U_w$  values of samples cooled in air and water after high-temperature exposure.

The  $U_w$  values of samples cooled in the air were higher than those cooled in water. This phenomenon has been expressed in similar studies in the literature as follows: The CH (calcium hydroxide) present in mortar samples undergoes dehydration and converts to CaO (calcium oxide) after high-temperature exposure. When the samples are cooled with water, CaO undergoes rehydration. Rehydration of CaO binds water and increases the mass of the sample. Therefore, when the samples are cooled with water, the mass losses are lower than in air cooling, resulting in higher  $U_w$  values [23]. The obtained results align with the study conducted by Alakara et al. [37].

#### 3.2. Ultrasonic Pulse Velocity (UPV)

## 3.2.1. The UPV Findings for Air-Cooled Samples

The UPV test is a nondestructive testing method used to assess the quality of concrete. It is sensitive to deterioration events in mortar samples, including internal cracking and other forms of degradation caused by thermal treatments [44]. The UPV values of samples cooled in the air after high-temperature exposure and their relative residual UPV values are presented in Figure 8. Upon examination of the UPV values, it can be observed that the UPV values of samples tested at laboratory conditions (20 °C) range from 4.40 km/s to 3.66 km/s. The quality of the samples tested in the laboratory environment is classified as "good", according to Whitehurst's classification provided in Table 3 [40]. The highest UPV values were obtained from samples coded as MP5, while the lowest UPV values were obtained from samples coded as MP25. After exposure to 300 °C temperature and subsequent air cooling, the UPV values of the samples increased compared with those of samples tested in laboratory conditions. The UPV values of samples exposed to 300 °C

and cooled in air ranged from 4.58 km/s to 3.88 km/s, with relative residual values ranging from 103.96% to 105.90%. As the temperature increased, the UPV values decreased significantly compared with those of samples tested in the laboratory environment. The relative residual UPV values of samples exposed to temperatures of 600 °C and 800 °C ranged from 68.10% to 71.47% and 26.84% to 31.86%, respectively. It is known that around 500 °C, calcium hydroxide (Ca(OH)<sub>2</sub>), a significant component of the hydration products of concrete/mortar, begins to break down, and the degradation of the C-S-H phase starts after 600 °C [36,45]. As a result, the mortar matrix acquired a porous structure, leading to a decrease in UPV values. Samples exposed to temperatures of 600 °C and 800 °C fall into the weak and very weak categories, respectively, according to the classification by Whitehurst [40].



**Figure 8.** The *UPV* values of air-cooled samples.

3.2.2. The UPV Findings for Water-Cooled Samples

The UPV values and relative residual UPV values of samples cooled with water after high-temperature exposure are presented in Figure 9. Upon examination of the UPV values, it can be observed that similar to the samples cooled in air, the highest UPV values were obtained from samples coded as MP5, while the lowest UPV values were obtained from samples coded as MP25. After high-temperature exposure, the UPV values of all samples decreased compared with those of samples tested at laboratory conditions. The UPV values of samples exposed to temperatures of 300 °C, 600 °C, and 800 °C and cooled with water ranged from 4.10 km/s to 3.41 km/s, 2.30 km/s to 1.80 km/s, and 1.25 km/s to 0.89 km/s, respectively. According to the classification by Whitehurst [40], the mortar samples exposed to temperatures of 300 °C, 600 °C fall into the "good," "weak," and "very weak" categories, respectively. The relative residual values for samples exposed to temperatures of 300 °C, 600 °C, and 800 °C ranged from 93.22% to 95.02%, 49.21% to 52.22%, and 24.31% to 28.33%, respectively. The sudden decrease in UPV values after temperatures exceed 300 °C can be explained by the breakdown of the hydration elements of cement and the rapid deterioration of the C-S-H phase due to sudden cooling. The decrease in UPV values



with an increase in the MP replacement ratio might be related to the insufficient hydration of MP beyond a certain replacement ratio [37].

Figure 9. UPV values of water-cooled samples.

3.2.3. Comparison of UPV Findings for Samples Cooled in Air and Water

The UPV results of samples cooled in air and water after high-temperature exposure are presented in Figure 10. The UPV values of samples cooled with water were lower than those of samples cooled in the air. This decrease was particularly noticeable in samples exposed to temperatures of 600 and 800 °C. The lower UPV values of samples cooled with water compared with those cooled in the air can be attributed to the rapid cooling of the samples with water after high-temperature exposure, leading to increased porosity and deterioration of the C-S-H phase [37]. Looking at the percentage change in UPV values between samples cooled in air and water, it can be observed that the UPV loss was at lower levels for samples exposed to 300 °C temperature. In the literature, it has been reported that samples exposed to temperatures around 200 °C experience a decrease in pore volume due to the evaporation of free water, leading to a strengthening of the composite [46,47]. The lower UPV losses for samples exposed to 300  $^{\circ}$ C temperature can be attributed to this phenomenon. It has been reported that microcracks begin to spread in cement paste and aggregates between 450 and 600 °C [48]. Topcu and Demir [49] have also reported that the breakdown of the C-S-H gel at temperatures above 450 °C reduces the speed of sound wave propagation and hence decreases UPV values. These conditions mentioned in the literature can explain the significant decrease in UPV values observed for samples exposed to temperatures of 600 and 800 °C. Overall, the UPV findings for samples cooled in air and water are consistent with the findings of previous literature studies [50,51]. Additionally, it has been reported that UPV results are related to pore structure and sample density and that there is a relationship between mechanical properties and UPV results [17,52].



Figure 10. The UPV values of samples cooled in air and water after high-temperature exposure.

#### 3.3. Compressive Strength

3.3.1. The  $f_{cs}$  Values of the Samples Air-Cooled after High-Temperature Exposure

The compressive strength ( $f_{cs}$ ) results of samples cooled in the air after exposure to high temperatures and the relative residual  $f_{cs}$  results compared with those of samples tested in the laboratory environment are presented in Figure 11. When examining the  $f_{cs}$ results of samples tested in the laboratory environment (20  $^{\circ}$ C), it can be observed that all mixture groups have  $f_{cs}$  values above the minimum  $f_{cs}$  value (42.5 MPa) specified in EN 196-1 [31]. The highest  $f_{cs}$  value was obtained from MP5-coded mortars. The  $f_{cs}$  value of MP5-coded mortars was 5.16% higher than that of Ref-coded mortars. With the increase in the MP replacement ratio, the  $f_{cs}$  values decreased. The strength loss that occurs with the increase in MP content can be attributed to the significant reductions in  $C_3S$  and  $C_2S$ , which are responsible for strength. This phenomenon is commonly known as the dilution of pozzolanic reactions. The obtained results are consistent with the literature [29,53]. When the mortar samples were cooled in the air after being exposed to 300 °C temperature, an increase in  $f_{cs}$  values between 3.50% and 8.98% was observed compared with those of samples tested in the laboratory environment. Alakara et al. [37] attributed this increase to the evaporation of free water in the cement paste. Dias et al. [54] attributed the increase in  $f_{cs}$  values at 300 °C to the thermal drying and dehydration of the cement paste. The obtained results are also consistent with the studies conducted by Yamanel et al. [29] and Alani et al. [55]. When the mortar samples were exposed to temperatures of 600  $^{\circ}$ C and 800 °C, significant decreases in  $f_{cs}$  values were recorded. The  $f_{cs}$  values of samples exposed to 600 °C temperature range between 24.29 and 33.79 MPa, while those of samples exposed to 800 °C temperature range between 12.86 and 18.17 MPa. The relative residual  $f_{cs}$  results for samples exposed to temperatures of 600 °C and 800 °C varied between 53.69% and 60.10% and 28.56% and 32.33%, respectively. The highest and lowest  $f_{cs}$  values for all temperature ranges were obtained from MP5- and MP25-coded mortars, respectively. The reason for the decrease in  $f_{cs}$  values as the temperature increases can be explained as follows: Around 550 °C, Ca(OH)<sub>2</sub>, which constitutes a significant portion of the hydrate products, starts to break down, and aggregates begin to deteriorate after this temperature [36,56]. At around 600 °C, the calcium silicate hydrate (C-S-H) phase degrades [57]. The C-S-H phase completely disintegrates above 800 to 900 °C [45,58].



**Figure 11.** The *f*<sub>*cs*</sub> values of air-cooled samples.

3.3.2. The f<sub>cs</sub> Values of the Samples Water-Cooled after High-Temperature Exposure

The  $f_{cs}$  values of the samples water-cooled after high-temperature exposure are presented in Figure 12.

When examining the  $f_{cs}$  results in Figure 12, it was noted that there was a significant decrease in  $f_{cs}$  values when the samples were cooled with water after exposure to high temperatures. The  $f_{cs}$  results of samples cooled with water after exposure to 300 °C temperature ranged between 38.45 MPa and 50.23 MPa. These samples' relative residual  $f_{cs}$  values ranged between 85.23% and 91.51%. Alakara et al. [37] suggested that the reason for this decrease in  $f_{cs}$  values could be due to the rapid reaction of CaO, which dissociates from Ca(OH)<sub>2</sub> after high-temperature exposure, with water, resulting in stresses and cracks in the cement paste. The  $f_{cs}$  values of samples cooled with water after exposure to temperatures of 600 °C and 800 °C were 19.52–25.23 MPa and 7.25–11.01 MPa, respectively. These samples' relative residual  $f_{cs}$  values ranged between 39.49% and 44.89% for 600 °C temperature and 15.46% and 19.59% for 800 °C temperature. For all temperature ranges, the highest and lowest  $f_{cs}$  values for samples cooled with water were obtained from MP5- and MP25-coded mortars, respectively. Similar decreases in  $f_{cs}$  values observed in samples cooled with water have also been reported in the studies by Yüzer et al. [59] and Karahan [60].



**Figure 12.** The *f*<sub>cs</sub> values of the water-cooled samples.

3.3.3. Comparison of  $f_{cs}$  Findings for Samples Cooled in Air and Water after High-Temperature Exposure

The  $f_{cs}$  findings for samples cooled in air and water after high-temperature exposure are presented in Figure 13.

When examining the effect of cooling regimes on strength losses, it was observed that the strength loss of mortars cooled with water is higher than that of those cooled in air. After exposure to temperatures of 300 °C, 600 °C, and 800 °C, the  $f_{cs}$  values of samples cooled with water showed decreases of approximately 12.89% to 20.54%, 19.64% to 30.25%, and 37.29% to 48.50%, respectively, compared with those of samples cooled in air. The decrease in  $f_{cs}$  values was particularly pronounced after exposure to 600 °C temperature. It is seen that the  $f_{cs}$  values of the 5%, 10%, and 15% MP-substituted mortars after being exposed to high temperatures of 300, 600, and 800  $^{\circ}$ C are higher than those of the Ref-coded mortars. Additionally, when the relative residual strength (RRS) graphs are examined, the RRS percentages of Ref-coded mortars are lower than those of MP-substituted mortars. This means that the strength loss of MP-substituted mortars is lower than that of reference mortars. This can be attributed to decreased  $Ca(OH)_2$  content in the mortar matrix as the MP substitution rate increases [37]. As mentioned before, the hydrated elements in the cement matrix break down after reaching 500 °C. As a result of the decrease in the density of the  $Ca(OH)_2$  structure, which is most damaged at high temperatures, in the mortar matrix, increases in RRS percentages were obtained as MP substitution increased. The findings obtained are also compatible with the literature [29]. Karahan [60] demonstrated that  $f_{cs}$  values of samples rapidly cooled with water are lower than those of samples cooled in air. In another study by Abadel et al. [61] investigating the effect of cooling regimes on  $f_{cs}$  values, higher rates of strength loss were observed in samples cooled with water. Yüzer et al. [59] suggested using alternative cooling methods instead of water for cooling samples after exposure to high temperatures.



Figure 13. The *f<sub>cs</sub>* findings for samples cooled in air and water.

## 3.4. Flexural Strength

3.4.1. The  $f_{fs}$  Values of the Samples Air-Cooled after High-Temperature Exposure

The flexural strength ( $f_{fs}$ ) findings for mortar samples cooled in the air after exposure to high temperatures and their relative residual  $f_{fs}$  findings compared with those of samples tested in the laboratory environment (20 °C) are presented in Figure 14. When examining the  $f_{fs}$  findings for samples tested in the laboratory environment (20 °C), it was observed that the highest  $f_{fs}$  value was obtained from MP5-coded mortars, and the lowest  $f_{fs}$  value was obtained from MP25-coded mortars. The  $f_{fs}$  value of MP5-coded mortars was 3.55% higher than that of Ref-coded mortars. Comparing the obtained  $f_{fs}$  findings with the literature, it has been reported that the  $f_{fs}$  values increase when MP is substituted up to a specific ratio, and the  $f_{\rm fs}$  results decrease with an increasing substitution ratio [29,62,63]. The results obtained are consistent with the literature. After exposure to 300  $^{\circ}$ C, the  $f_{fs}$ values of samples cooled in air ranged between 9.01 MPa and 10.36 MPa, and these values showed an increase of approximately 3.24% to 6.50% compared with those of samples tested in the laboratory environment. The increases observed here exhibited a similar trend to the increase in  $f_{cs}$  values. As mentioned in the section interpreting the  $f_{cs}$  findings, the increase in  $f_{fs}$  values can also be attributed to the evaporation of free water in the cement paste. A similar phenomenon has been noted in a study by [35] Sarıdemir and Yıldırım [64]. As the temperature to which the samples are exposed reached 600  $^{\circ}$ C and 800  $^{\circ}$ C, the  $f_{fs}$ values of the samples ranged between 4.45 MPa and 5.42 MPa and 1.19 MPa and 2.24 MPa, respectively. The relative residual  $f_{fs}$  results for samples exposed to temperatures of 600 °C and 800 °C varied between 52.60% and 54.70% and 14.07% and 22.60%, respectively. The decrease in  $f_{fs}$  values of samples exposed to temperatures above 600 °C can be attributed to the breakdown and deterioration of C-S-H, which is one of the hydration products, different expansions of aggregate and paste, excessive shrinkage, and the disintegration of the interfacial zone [65,66].



**Figure 14.** The  $f_{fs}$  values of the air-cooled samples.

3.4.2. The  $f_{fs}$  Values of the Samples Water-Cooled after High-Temperature Exposure

The  $f_{fs}$  findings for samples cooled in water after exposure to high temperatures and their relative residual  $f_{fs}$  findings are presented in Figure 15. When samples exposed to high temperatures were cooled in water, significant decreases in  $f_{fs}$  values were observed. The  $f_{fs}$ values of samples cooled in water exhibited a similar trend to the decrease in  $f_{cs}$  values for these samples. After exposure to 300 °C, the  $f_{fs}$  values of samples cooled in water ranged between 6.87 MPa and 9.23 MPa, and their relative residual  $f_{fs}$  values showed a decrease of approximately 18.79% to 2.91% compared with those of samples tested in the laboratory environment. The reason for this decrease can be attributed to the rapid cooling of samples after high-temperature exposure, resulting in additional stresses and cracks in the cement paste, as mentioned in the section interpreting the  $f_{cs}$  findings [37]. The decreases in  $f_{fs}$ values were particularly pronounced at temperatures above 300 °C, which could result from the decomposition of hydrated products at higher temperatures [67]. Samples exposed to 600 °C and 800 °C were determined to exhibit  $f_{f_{s}}$  values ranging between 2.94 MPa and 4.86 MPa and 1.08 MPa and 1.98 MPa, respectively. Their relative residual  $f_{fs}$  values for temperatures of 600  $^\circ$ C and 800  $^\circ$ C varied between 34.75% and 49.04% and 14.11% and 19.98%, respectively. Similar decreases in  $f_{fs}$  values for mortar samples cooled in water have been reported in a study by Karahan et al. [23].



**Figure 15.** The *f*<sub>*fs*</sub> values of the water-cooled samples.

3.4.3. Comparison of  $f_{fs}$  Findings for Samples Cooled in Air and Water

The  $f_{fs}$  values of samples cooled in air and water are presented in Figure 16. When the effect of cooling regimes on  $f_{fs}$  findings is evaluated, it was determined that the strength loss of mortar samples cooled in water was higher than in air. A similar situation was observed in the  $f_{cs}$  values as well. After exposure to 300 °C, 600 °C, and 800 °C, the  $f_{fs}$  values of samples cooled in water decreased by approximately 7.49% to 23.75%, 10.35% to 33.93%, and 6.40% to 24.88%, respectively, compared with those of samples cooled in air. A study by Karahan et al. [23] investigating the effect of the cooling regime found that the loss of  $f_{fs}$  in samples cooled in water is higher than that of those cooled in air. A similar observation was reported by Husem [68]. When samples are rapidly cooled in water, the additional stresses and cracks formed in the cement paste matrix result in lower  $f_{fs}$  values for these samples than those cooled in air.

#### 3.5. Mass Loss

# 3.5.1. The Mass Loss Values of the Air-Cooled Samples

The weight losses of samples exposed to high temperatures and cooled in air are presented in Figure 17. It was observed across all mixture groups that as the temperature increases, the weight losses also increase. However, as the mixtures' MP substitution ratio increases, weight loss decreases were identified. The weight losses of samples exposed to 300 °C, 600 °C, and 800 °C and cooled in air varied between 3.58% and 4.65%, 8.73% and 9.96%, and 14.73% and 15.96%, respectively. When the weight losses of MP25-coded mortars at 300 °C, 600 °C, and 800 °C were compared with those of Ref-coded mortars, reductions of 23.01%, 12.35%, and 7.71% were observed, respectively.



**Figure 16.** The  $f_{fs}$  findings for samples cooled in air and water.



Figure 17. The mass loss values of the air-cooled samples.

The weight loss increased with temperature. Generally, the weight loss occurring when mortar specimens are exposed to temperatures around 200  $^{\circ}$ C is due to the evaporation of water that can vaporize and the release of free water in capillary pores. The weight loss around 400  $^{\circ}$ C results from the decomposition of carboaluminate hydrates, which eliminates water in chemical bonds. At 400  $^{\circ}$ C, all capillary water in the mortar matrix is lost.

When the temperature exceeds 600 °C, breakdowns in the C-S-H gels are observed. When mortar specimens are subjected to high temperatures, hydration products like calcium hydroxides are initially affected, followed by the degradation of C-S-H gels [37]. Using mineral additives in cementitious composites has been reported to increase resistance to high temperatures as these additives convert calcium hydroxides into C-S-H gels, which would be the first to deteriorate [69].

## 3.5.2. The Mass Loss Values of the Water-Cooled Samples

The mass losses of water-cooled samples exposed to high temperatures are presented in Figure 18. When the specimens are cooled with water after exposure to high temperatures, an increase in weight loss, similar to what was observed in air-cooled specimens, can be seen. For all temperature levels, the highest weight loss was observed in the Ref-coded mortars, while the lowest was in the MP25-coded mortars. After exposure to temperatures of 300 °C, 600 °C, and 800 °C, the weight losses of water-cooled specimens ranged from 3.19% to 4.13%, 8.35% to 9.49%, and 13.63% to 14.91%, respectively. When comparing the weight losses of MP25-coded mortars at 300 °C, 600 °C, and 800 °C with those of the Ref-coded mortars, reductions of approximately 22.76%, 12.01%, and 8.58% were observed, respectively. In conclusion, as with the air-cooled specimens, it was determined that an increase in the MP substitution ratio leads to decreased weight losses.



Figure 18. The mass loss values of the water-cooled samples.

3.5.3. Comparison of Mass Loss Findings for Samples Cooled in Air and Water

The mass losses of specimens cooled in air and water are shown in Figure 19. Upon examining the values in the graph, it can be observed that the mass losses of specimens cooled in water are lower than those of specimens cooled in air. The mass losses of specimens cooled in water, compared with those of specimens cooled in air, are lower by percentages ranging from 8.33% to 11.18%, 4.25% to 5.33%, and 5.67% to 7.47% for temperature values of 300 °C, 600 °C, and 800 °C, respectively.

The lower mass losses of specimens cooled in water compared with those of specimens cooled in air can be attributed to the fact that the cementitious matrix regains some of the lost water during the water-cooling process. Karahan et al. [23] explained this phenomenon by stating that the rehydration of CaO formed due to the dehydroxylation of CH (calcium hydroxide) in the cementitious matrix after high-temperature exposure can occur during

water cooling. Consequently, when the specimen is cooled in water, CaO is hydrated by water, and the rehydration of CaO binds water, increasing the specimen's mass. The observation that the mass losses of specimens cooled in water are lower than those of specimens cooled in air aligns with findings from previous studies in the literature [50,70].



Figure 19. The mass loss findings for samples cooled in air and water.

# 3.6. Microstructural Analyses

Microstructural analyses of the Ref- and MP10-coded specimens after testing in the laboratory environment (20 °C) and at a high temperature of 600 °C were carried out using scanning electron microscopy (SEM). SEM images of the Ref- and MP10-coded specimens tested in the laboratory environment are presented in Figures 20a and 20b, respectively. When comparing the SEM images of the Ref- and MP10-coded specimens, it can be observed that the Ref-coded specimens have a higher density of C-S-H (calcium silicate hydrate) and a greater number of hydrated elements. C-S-H gels cover the entire surface in a network-like structure in the Ref-coded specimens. On the other hand, the C-S-H density in the MP10-coded specimens is lower. This phenomenon can be associated with the slower development of hydration in pozzolanic materials [71].



Figure 20. SEM images of samples in a laboratory environment. (a) Ref, (b) MP10.

The SEM images of the Ref- and MP10-coded specimens after they were subjected to the high temperature of 600 °C and air-cooled are presented in Figures 21a and 21b, respectively. When comparing these SEM images with the ones shown in Figure 20, it can be observed that the C–S–H gel structure deteriorated after the high-temperature exposure, which is particularly evident in Figure 21b, where it can be seen that microcracks have formed. These SEM images confirm the deterioration in mechanical properties. It can be noted that the C–S–H gel structure in Figure 21a is relatively better preserved compared with that in Figure 21b. Additionally, microcracks are more clearly visible in Figure 21b.



**Figure 21.** SEM images of samples cooled in air after 600 °C high-temperature exposure. (**a**) Ref, (**b**) MP10.

The SEM images of the Ref- and MP10-coded specimens after they were exposed to the high temperature of 600 °C and cooled with water are provided in Figures 22a and 22b, respectively. When comparing these images in Figure 22 with the SEM images of the specimens tested in the laboratory environment shown in Figure 20, it can be stated that the C–S–H gel structure significantly deteriorated after high-temperature exposure, with an increase in microcracks and pore density. When comparing the SEM images of specimens cooled with water (Figure 22) with those of specimens cooled in the air (Figure 21), it is evident that the C–S–H structure is further damaged in the specimens cooled with water, and the number of pores and crack widths in the specimen matrix have increased. In conclusion, it can be said that the specimens exposed to high temperatures and cooled with water are more deformed than those cooled in the air.



**Figure 22.** SEM images of samples cooled in water after 600 °C high-temperature exposure. (**a**) Ref, (**b**) MP10.

# 4. Conclusions

This study investigates the effects of high temperature and cooling regimes on the physical, mechanical, and microstructural properties of cementitious composites containing 5%, 10%, 15%, 20%, and 25% replacements with marble powder (MP). The cementitious composites were subjected to measurements of water absorption ( $U_w$ ), ultrasonic pulse velocity (UPV), compressive strength ( $f_{cs}$ ), flexural strength ( $f_{fs}$ ), and mass loss after exposure to high temperature. Microstructural analyses were also conducted using scanning electron microscopy (SEM). The obtained results from the study are as follows:

- With an increase in the replacement percentage of MP,  $U_w$  values decreased. This can be attributed to the lower specific gravity of MP compared with cement. The  $U_w$  values of specimens cooled in water were higher than those of specimens cooled in air. This is because, after high-temperature exposure, the dehydration of calcium hydroxide (CH) results in its conversion to CaO, which undergoes rehydration during water cooling.
- UPV values decreased with an increase in the MP replacement percentage. This decrease might be attributed to reduced hydrated elements due to higher MP content, leading to a more porous cement matrix. The UPV values of specimens cooled in air are higher than those of specimens cooled in water. Rapid water cooling induces additional stress and cracks in the cement matrix, making the specimens more porous. Consequently, the UPV values of specimens cooled in water are lower than those of specimens cooled in air.
- The results obtained from  $f_{cs}$  and  $f_{fs}$  showed a similar trend. The best mechanical properties were observed in specimens with 5% MP replacement. As the replacement percentage increases, a deterioration in mechanical properties is observed. The  $f_{cs}$  and  $f_{fs}$  values of specimens cooled in water were lower than those of specimens cooled in air. This deterioration became more pronounced with an increase in the exposure temperature. The reduced mechanical properties of specimens cooled in water can be attributed to the additional stress and cracks induced by rapid cooling.
- The mass losses of all specimens increased with higher temperatures. This is attributed to the breakdown of hydrated elements with increasing temperature. An increase in the MP replacement percentage results in decreased mass losses. The mass losses of specimens cooled in air were higher than those of specimens cooled in water. This is due to the dehydration of CH into CaO after temperature exposure, followed by rehydration of CaO during water cooling. Rehydrated CaO absorbs water, increasing the mass of the specimen.

In conclusion, using MP in cementitious mixes significantly enhanced the physical, mechanical, and microstructural properties of cementitious materials exposed to high temperatures. As the MP replacement percentage increases, notable reductions in mass loss are identified. Furthermore, not only does the substitution of MP for cement have the potential to mitigate environmental pollution associated with MP disposal and lead to savings in cement consumption, it is also anticipated that using MP as a substitute for cement could decrease CO<sub>2</sub> emissions and energy costs. Future research could explore the effects of alternative cooling methods beyond air and water cooling on MP-incorporated mixes exposed to high temperatures. In another study, the effect of MP particle size on the strength and durability properties of cementitious composites exposed to high temperatures can be examined. Decreasing the particle size would improve hydration and significantly contribute to the strength properties of the composites.

**Author Contributions:** Conceptualization, O.S. and I.D.; methodology, O.S. and I.D.; validation, O.S.; investigation, O.S. and İ.R.B.; resources, O.S. and İ.R.B.; writing—original draft preparation, O.S. and İ.R.B.; writing—review and editing, O.S., I.D. and İ.R.B.; supervision, O.S. and I.D. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

**Conflicts of Interest:** The authors declare no conflict of interest.

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