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Influence of Various Heat Treatment Regimes on Properties of Concrete Incorporating Marble Waste Powder as Cement Replacement

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ABSTRACT

This work investigates the influence of thermal curing regimes on the properties of concrete incorporating marble waste powder. To achieve this objective, 6%, 12%, and 18% of marble waste powder were introduced in concrete composition as a cement replacement. Then, concrete mixtures prepared were cured at temperatures of 25℃, 45℃, 65℃, and 85℃ for 24 and 48 hours, respectively, to investigate the effect of these thermal treatments on the mechanical strengths of concrete mixtures. The durability properties were investigated through the water absorption, ultrasonic pulse velocity, and concrete resistance against HCl and H_2SO_4 ions attack. The results obtained showed that thermal treatment enhances the mechanical properties of concrete at an early age but tends to decrease them over the long term. At 2 days, the compressive strength increased by 48.54 and 24.82% for concretes containing 0 and 18% of marble waste powder and cured at 45℃ for 24 hours, while at 85℃, the compressive strength of these mixtures decreases by 17.06% and 6.67%. The flexural strength also increases by 54.84% for concrete modified with 12% of marble powder and cured at 45℃ for 24 hours. Furthermore, the increase in the curing time from 24 to 48 hours negatively affects the compressive strength of concretes modified with marble waste powder. The results obtained also showed that the increase in the temperature to 85℃ increases the water absorption by 0.99% and decreases ultrasonic pulse velocity by 9.04%. Furthermore, concrete treatment at 45℃ is found to be the optimum regime for concrete resistance to the acid attack, while the incorporation of marble powder mitigates the adverse effects of the higher temperature treatment.

1. INTRODUCTION

Over several decades, concrete has been considered as the most widely used materials in construction. However, its strength and durability can be affected by various environmental factors such as temperature and humidity. To enhance the mechanical properties of concrete, various thermal treatment methods have been developed [1].

It was demonstrated that concrete specimens subjected to an early high temperature of 40℃ had higher initial strength by 72.73% compared to concrete cured at 20℃, but exhibited lower strength at later stage. Conversely, specimens subjected to early low temperature (5℃) show lower initial strength but develop aging resistance almost equivalent to that of specimens subjected to isothermal curing (20℃) [2]. Kim et al. [3] investigated the effect of curing temperature, aging, and cement type on concrete mechanical properties. They found that concrete exposed to high temperature during early curing stages exhibited higher compression and flexural strength at early age but lower compression and flexural strength at older age. The curing temperature effect on the modulus of elasticity was not as significant as on the compression strength. This trend was also observed by Topcu and Toprak [4]. Singh et al. [5] conducted a technical study aimed to increasing the curing rate of concrete. They concluded that high-temperature curing of concrete was highly beneficial, while the acceleration through the use of accelerators admixtures can lead to many potential problems and difficulties. Hwang et al. [6] demonstrated a significant advantage of steam curing at 60℃. Through this method, concrete can develop compression strength six times higher than the same concrete when it was cured for 18 hours at 60℃. Liu et al. [7] found that steam curing thermal treatment of concrete at 60℃ increased the capillary absorption and concrete sorptivity coefficient. Zeyad et al. [8] found that thermal treatment of concrete at a temperature exceeding 80℃ has a negative influence on the microstructure and strength of concrete at long-term. Liu et al. [9] investigated the concrete mechanical properties modified with fly ash and subjected to steam curing at high temperatures of 60℃. They reported that for 13 hours of curing, concrete with fly ash exhibited low compressive strength by 19.08% at early age and after 28 days. Gesoglu [10] studied the influence of steam curing acceleration at 70℃ on concrete incorporating metakaolin and silica fume. He found that steam-cured concrete at high temperatures showed higher compression strength at an early age by 10%, while from 7 days onward, this concrete showed lower strength. Jacquemot et al. [11] studied the effect of temperature treatment on self-compacting concrete properties. They observed that high-temperature curing at 65°C had a positive effect on the compressive strength which increased by 23.08% at 28 days. Bingöl and Tohumcu [12] found that steam treatment of self-compacting concrete incorporating silica fume and fly ash at 70°C for 16 hours can achieve compression strength equivalent to 95% of that of standard cured concrete samples. Derabla and Benmalek [13] studied the behavior of self-compacting concrete thermally treated and modified with granulated slag, fine limestone, and crystallized slag. They found that the effect of thermal treatment at 60°C for 24 hours was very pronounced at early age, as all thermally treated self-compacting concretes achieved higher strength by 156% than untreated self-compacting concretes. However, a minimal strength loss by 32% was observed in the long term. Ramezanianpour et al. [14] demonstrated that the increase in the initial compressive strength of self-compacting concrete thermally treated at 60-70°C was accompanied by an increase in the permeability. Derabla and Sajed [15] studied the properties of self-compacting mortar thermally treated at 60°C. They found that the thermal treatment can improve the compressive strength. Additionally, they observed that the incorporation of limestone filler and ground granulated blast furnace slag enhanced the durability of self-compacting mortar subjected to thermal treatment. Kang et al. [16] studied the effect of thermal treatment on high-performance concrete. They found that this treatment method had a positive effect on the hydration of such concrete. They also observed that thermal treatment at high temperatures ensures exceptional strength at early age. However, there was no increase in long-term strength; instead, a slight decrease was observed. Moreover, long-term flexural and tensile strengths were similar regardless of curing conditions. Similar finding was noted by Fladr and Broukalova [17]. Shen et al. [18] added that the pores in high-performance concrete increased when it was exposed to thermal treatment. Mo et al. [19] found that steam thermal treatment increased the compressive strength at early age and significantly improved the reaction of metakaolin in the matrix of ultra-high-performance concrete. Stind et al. [20] found that increasing thermal treatment time to 24 hours reduced the shrinkage stresses of high-performance concrete. Zeyad et al. [21] and Rahimov and Muminov [22] studied the influence of thermal treatment on the properties of high-strength concrete containing varying amounts of ultrafine palm oil fuel ash. They found that this treatment was beneficial only at early age.

On the other hand, several researchers worked on the incorporation of industry waste in concrete composition to reduce the higher consumption of natural resources. Marble waste (MW) was subjected to several studies during the last decade [23] to investigate the possibility of its use as powder or aggregates. Djebien et al. [24] found that adding marble waste powder (MWP) to sand concrete composition significantly improved its workability. This trend was also observed by Rashwan et al. [25], who used MWP as a partial substitute for cement. This trend was explained by the fact that MWP minimize the friction between the constituents and facilitates concrete flow. Pal et al. [26] substituted cement with MWP, finding that compressive strength increased with increasing MWP content up to an optimal value corresponding to 10%. The gain in compressive strength was 8.54% and 12.84% at 7 and 28 days, respectively. This trend was observed and explained by the filling effect of MWP which increases concrete compactness [27, 28]. Rana et al. [29] observed that adding MWP to concrete composition reduced compressive strength values regardless of the MWP content. This reduction becomes significant when the substitution rate exceeds 10%. Topçu et al. [30] used MWP in self-compacting concrete formulation, concluding that MWP significantly reduced compressive strength when the substitution rate exceeded 200 kg/m^3. Djebien et al. [31] studied the incorporation of MW as fine aggregate in self-compacting concrete formulation. They observed that the introduction of MW as sand decreased the air content and reduced the compressive strength. Tennich et al. [32] introduced MWP in concrete composition by substituting cement, noticing a decrease in the modulus of elasticity. Hameed and Sekar [33] studied the durability of MWP-based concrete, noting that concrete modified with MWP had higher sulfate ions resistance than concrete without MWP. Binici et al. [34] found that adding MWP to concrete composition improved concrete resistance to sulfate ions and reduced chloride ions penetration. Ince et al. [35] observed that MWP-based concretes had good resistance to freeze-thaw phenomenon. Monica and Dhoka [36] noted that water absorption of concrete modified with MW is slightly higher than that of ordinary concrete.

2. RESEARCH SIGNIFICANCE

During the last decades, several studies were carried out to investigate the effect of the temperature treatment on properties of different types of concrete containing mineral additions such as fly ash, limestone filler, silica fume, and ground blast furnace slag. They highlighted the improvement of concrete mechanical properties with thermal treatment, particularly in early age. However, no relevant work has been carried out to study the effect of MWP as cement replacement on the concrete properties. This work fills this gap through the investigation of the temperature treatment effect at 45°C, 65°C, and 85°C on the mechanical strengths and durability of concrete containing 6%, 12%, and 18% of MWP as a cement replacement. Mechanical properties were evaluated by the compressive strength and flexural strength, while durability properties were investigated through the water absorption, ultrasonic pulse velocity, and chemical acid attack tests.

3. MATERIALS USED AND TESTS

In this experimental work, Portland cement CEM II 42.5 (PC) was used. Tables 1 and 2 show the properties of cement used. This cement was used by Djebien et al. [23]. Crushed limestone fine aggregate (CFA) with a maximum nominal size of particles of 4 mm was used in this study as sand (CS). As coarse aggregates (CCA), two gravels (G1 and G2) having nominal size of 16 mm and 25 mm were used, respectively.

The properties of the CFA and CCA are presented in Table 3. Furthermore, Figure 1 depicts the particle size distribution of aggregates used.

Marble powder (MWP) was collected in wet form from the marble quarry of Skikda (Eastern Algeria), and was prepared by air drying followed by oven drying at 105°C, to obtain finely ground dry powder with a specific surface of 2608 cm^2/g and a particle size less than 80 µm. Table 2 shows the chemical composition of MWP [23]. A superplasticizer (SP) based on poly-carboxylate was used. This was POLYFLOW SR 5400 having brown color, a pH of 5, and a density of 1.07.

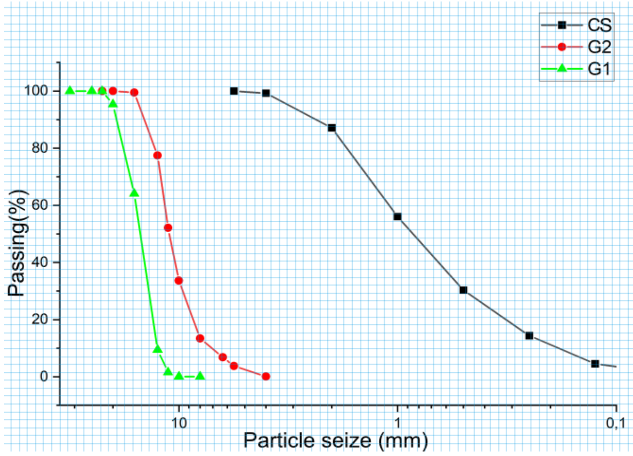


Figure 1. Particles size distribution of aggregates

Table 1. Properties of PC [23]

Properties	PC
Initial setting time (min)	75
Final setting time (min)	181
Specific gravity	3.1
Blaine specific surface (cm^2/g)	3156
Compressive strength for 2 days (MPa)	26.28
Compressive strength for 7 days (MPa)	30.57
Compressive strength for 28 days (MPa)	43.95
Flexural strength for 2 days (MPa)	6.09
Flexural strength for 7 days (MPa)	7.89
Flexural strength for 28 days (MPa)	10.47

Table 2. Chemical composition of PC and MWP [23]

Chemical Properties (%)	PC	MWP
CaO	61.38	53.85
Al_2O_3	5.18	0.38
Fe_2O_3	3.34	0.22
SiO_2	19.97	1.11
MgO	0.99	2.81
Na_2O	0.2	0.15
K_2O	0.44	0.04
Cl-	0.19	0.02
SO_3	2.90	4
Loss in ignition	3.71	-
C_4AF	10.25	-
C_3A	8.08	-
C_3S	58.45	-
C_2S	13.24	-

Table 3. Properties of aggregates

Properties	CCA		CFA
	G1	G2	CS
Specific gravity (g/cm^3)	2.74	2.65	2.61
Sand equivalent (%)	-	-	68.4
Finenesmodulus	-	-	2.61
Methylene blue value	-	-	0.5
Micro-deval (%)	17.90	15.60	-
Los Angeles (%)	22.08	20.10	-
Absorption (%)	0.72	0.60	1.88

Table 4. Concrete mixtures

	MC	CMW6	CMW12	CMW18
G1 (kg/m^3)	386	386	386	386
G2 (kg/m^3)	578	578	578	578
CS (kg/m^3)	898	898	898	898
PC (kg/m^3)	350	328.98	307.96	286.94
SP (l/m^3)	3.675	3.675	3.675	3.675
Water (l/m^3)	184	184	184	184
MWP (kg/m^3)	0	19.65	39.29	58.94
Slump (cm)	18.5	19.6	20.7	21.5
Fresh density	2.43	2.37	2.34	2.29

The Dreux-Gorisse method was used for the composition of control concrete (MC). Then, a substitution of 6% (CMW6), 12% (CMW12), and 18% (CMW18) of the total PC volume with MWP was carried out, without changing the content of CCA, CFA, water, and SP, which were remained constant (Table 4). These substitution rates of MWP were selected based on the previous studies which noted that a substitution rate not exceeding 10-15% does not significantly affect the compressive strength of concrete. The SP content was 1% of the cement weight. The solid ingredients were mixed for three minutes to ensure the homogeneity of mixtures. Then, superplasticizer and water were added and mixed for other three minutes with the other constituents according to NF P18-405.

The fresh mixtures were poured and vibrated into molds with dimensions of (150×150×150 mm^3), (100×100×100 mm^3), and (70×70×280 mm^3). After placing the fresh concrete into the molds and vibrating it, the samples are positioned in a curing oven at designated temperatures for varying durations. The curing temperature was set at three levels, 45°C, 65°C, and 85°C, and the curing durations for these temperatures were set at 24 hours and 48 hours, respectively. After the end of the curing period, the samples were demolded and kept in a water basin at a temperature of 25°C until the test date.

The cube samples of (150×150×150 mm^3) were used for water absorption, ultrasonic pulse velocity, and compressive strength tests in accordance with NF EN 12390-3, NF EN 12504-4, and NBN B 15-215 standards, respectively. The prismatic samples of (70×70×280 mm^3) were used for flexural strength according to NF EN 12390-5 standard. The cubic samples of (100×100×100 mm^3) were used for durability tests using ASTMC 267-96 standard. After the initial thermal treatment, the specimens continued their mature in a water basin maintained at a temperature of 25°C. After 28 days of curing, the samples of durability tests were divided into two distinct groups, and then immersed in containers containing chemical solutions, namely hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) with a concentration of 5%. The samples remained in these containers until the test days at 56 days, 90 days, and 180 days. A pH meter was used to monitor the pH level weekly.

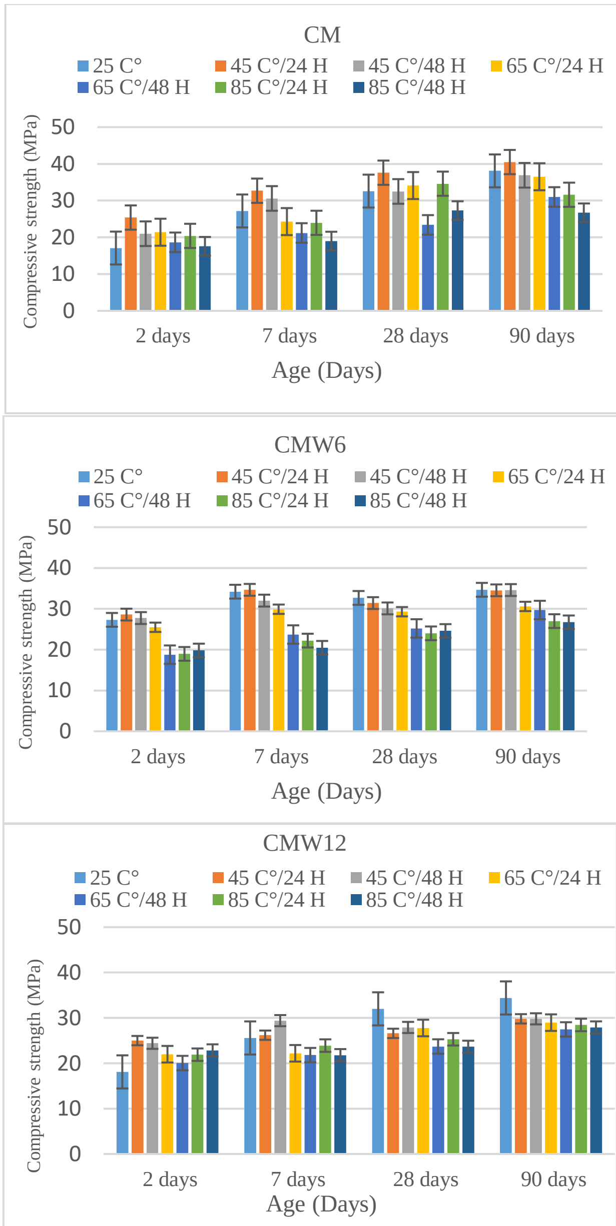
4. RESULTS AND DISCUSSION

4.1 Compressive strength

Figure 2 depicts the compressive strength of concrete incorporating MWP under various curing regimes. It's notable that the addition of 6%MWP increases the compressive strength at early ages of concrete, particularly at 2 days, aligning with the results found by Shirule et al. [27] and Ergün

[28]. This trend is explained by the filling effect of the MWP fines, enhancing the interfacial transition zone (ITZ) properties surrounding the aggregates. However, in the long term, MWP-based concretes exhibit lower strengths compared to the control concrete. This reduction is more significant with 18% of MWP. This trend was reported by Aliabdo et al. [37]. This is likely due to the reduction of cementitious material (C3S and C2S), which primarily contributes to concrete strength.

The results indicate that concrete samples subjected to thermal treatment show an increased compressive strength at early ages. This increase is particularly significant for treatment at 45℃ for 24 hours, reaching an increase by 48.54% for (CM) mixture. However, it's essential to note a decrease in the strength of samples subjected to 48 hours thermal treatment with a reduction of 29.92% observed for (CM) mixture treated at 85℃ for 48 hours. This trend has been previously observed [38, 39] and attributed to the fact that the increase in heat treatment duration negatively affect the quality and the orientation of hydrates, and increase the shrinkage values of concrete.



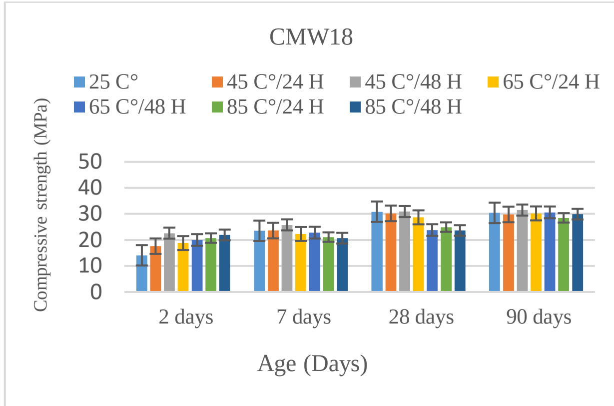


Figure 2. Compressive strength of concrete mixtures

The increase in concrete compressive strength during early thermal treatment is attributed to enhanced hydration reaction, resulting in higher strength gain. High-temperature curing also increases the reactivity of C3S, leading to a higher strength gain at early stages [7, 40], while the decrease in long-term compressive strength of concrete during high-temperature curing could be attributed to non-uniform distributions of hydrates, resulting in a less dense microstructure compared to curing under moist conditions. This observation has been confirmed by [10, 41]. It is also observed that concrete incorporating MWP and subjected to 45℃ exhibits higher compressive strength values at early ages, with a significant increase as the content of MWP is increased. The compressive strength at 2 days of CMW18 mixture treated at 45℃ for 48 hours increases by 60.28% compared to the control concrete. At 90 days the addition of MWP appears to reduce the compressive strength losses after thermal treatment, reaching only 1.64% for treatment at 85℃ for 48 hours. In contrast, control concrete treated in the same manner show strength loss up to 29.92%. The increase in compressive strength of concrete modified with MWP is attributed to the nucleation effect of MWP which improve the kinetic of hydration and the quality of hydration products and lead to increase the compressive strength at early age [42].

4.2 Flexural strength

According to Figure 3, a decrease in flexural strength is evident upon replacing cement with MWP. This decrease is more pronounced at early ages, reaching 50% for 18% replacement rate. This trend has been observed by Aliabdo et al. [29]. This is likely attributable to the reduction of cementitious material (C_3S and C_2S), which contribute to the concrete strength. The results obtained demonstrate that thermal treatment at 45℃ for 24 hours increases early-age flexural strength. The increase in flexural strength reaches 6.78% at 7 days for (CM) mixture. Conversely, there is a decrease in flexural strength observed with higher temperatures and longer curing periods. Flexural strength losses reach 50% for (CM) mixture subjected to treatment mode at 85℃ for 48 hours. It is noted that the incorporation of MWP enhances the flexural strength of concrete samples subjected to thermal treatment, particularly at early ages. This enhancement reaches a maximum increase by 54.84% for the treatment at 45℃ for 24 hours, for concrete modified with 12% MWP.

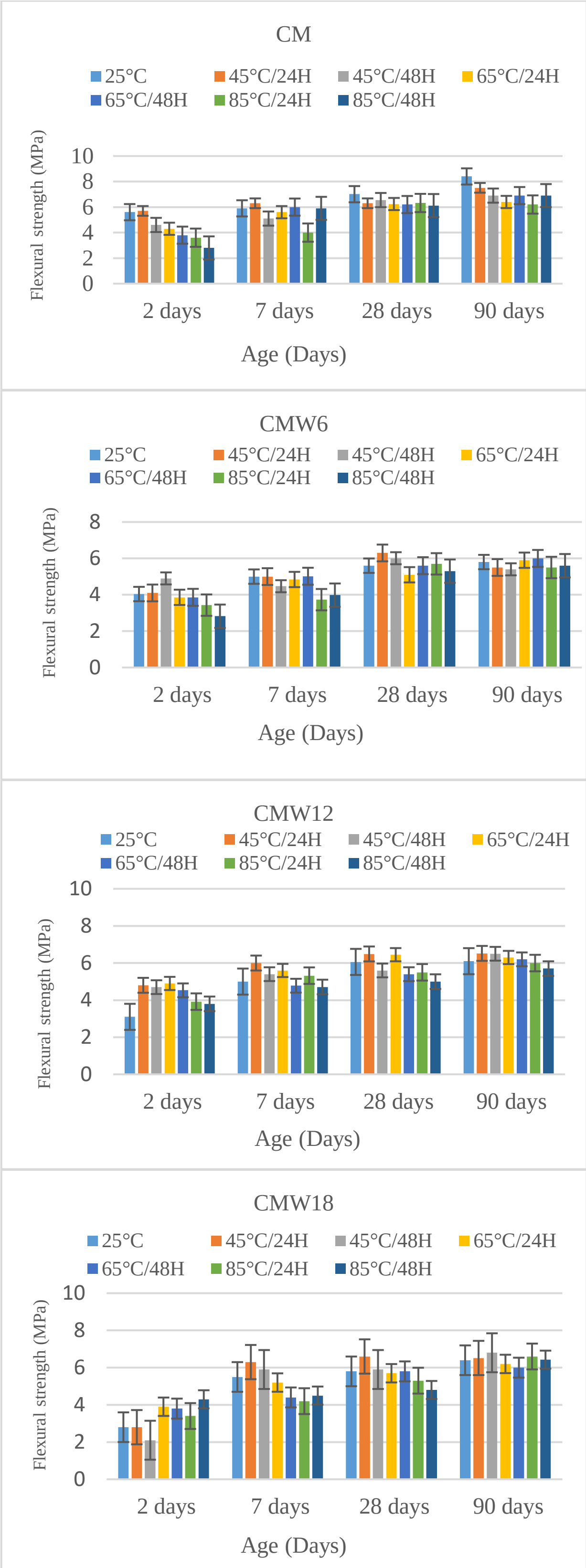


Figure 3. Flexural strength of concrete mixtures

4.3 Ultrasonic pulse velocity (UPV)

The variations in ultrasonic pulse velocity of concrete mixes at 28 days are illustrated in Figure 4. A decrease of 1.45% in UPV is observed for concrete containing 6% MWP. Furthermore, as the MWP content increases, the decrease in UPV becomes more significant, reaching 2.46% for concrete containing 18% MWP. It can be also seen that UPV decreases when the time and temperature of curing increased. This decrease reaches 9.04% for the treatment mode at 85°C for 48 hours for CM mixture. The reduction in UPV of thermally treated samples is attributed to the increase in their porous structures, which affect the propagation of UPV through the concrete. This trend has been observed by Derabla and Benmalek [13].

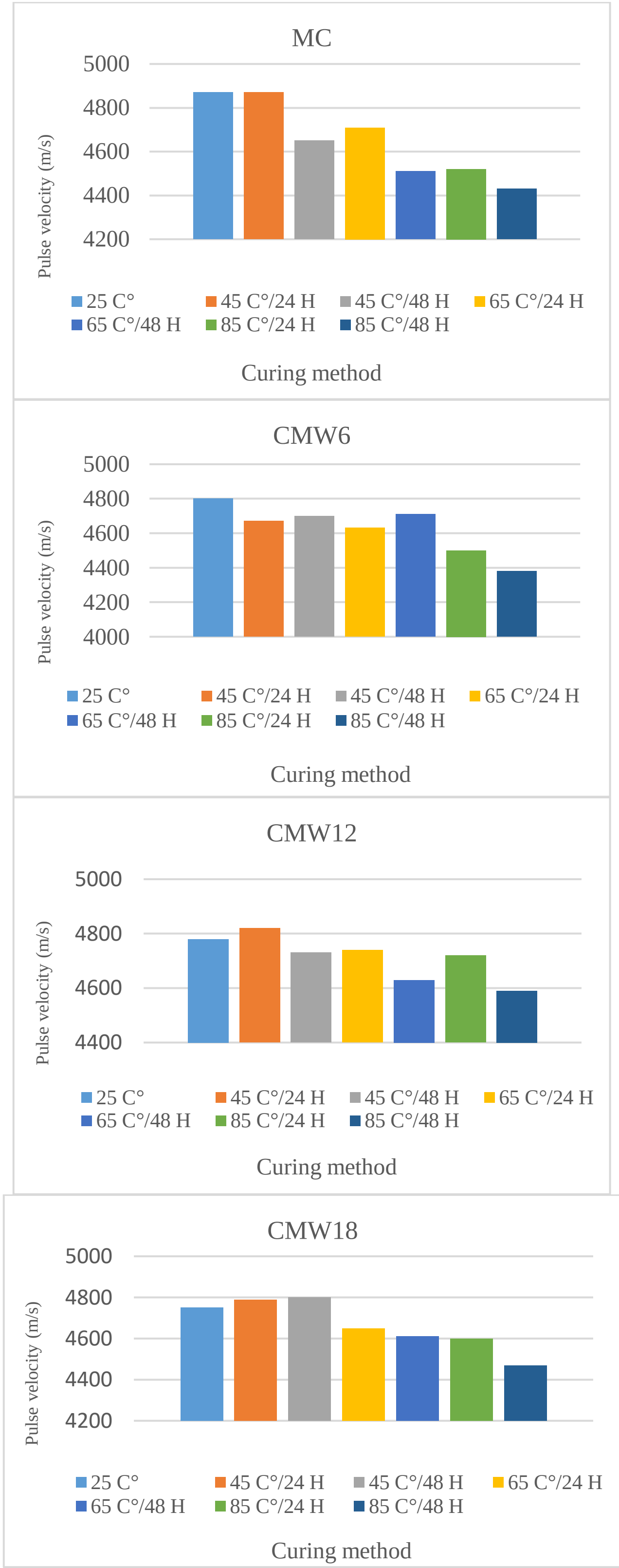


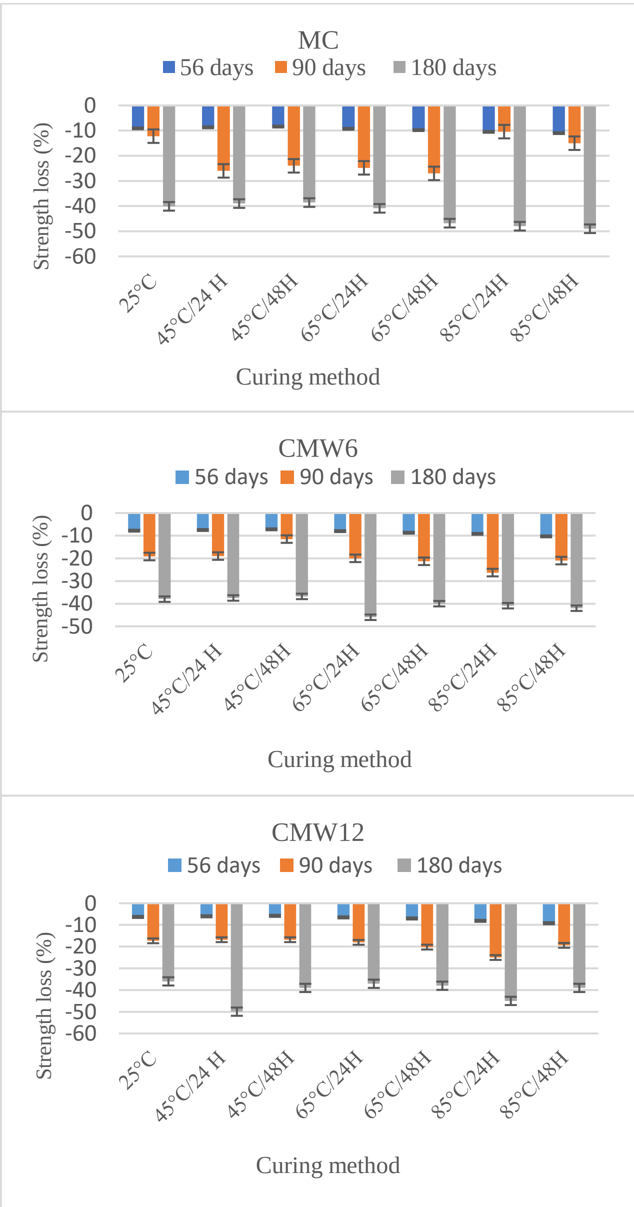
Figure 4. Pulse velocity values of concrete mixtures

4.4 Chemical attack by HCl

4.4.1 Compressive strength loss

Figure 5 demonstrates that MWP plays a positive role in the resistance against HCl ions. The incorporation of MWP gradually decreases the compressive strength loss. At 25℃, it can be observed that the control concrete undergoes a strength loss by 9.1%, while this loss decreases to 5.9% when MWP content increased to 18%. This is attributed to the improvement of concrete compactness as reported [43, 44]. It can also be shown that the resistance of concrete to HCl is enhanced at 45℃. When the temperature exceeds 45℃, the strength loss becomes significant. The final loss reaches 49% for the control concrete mixture treated at 85℃ for 48 hours. This increase in compressive strength loss is attributed to the increase in porosity and water absorption of thermally treated concrete at higher temperature that favorites the penetration of ions in the concrete.

The introduction of MWP in concrete composition appears to mitigate the negative effects of thermal treatment on concrete HCl resistance. Indeed, the strength loss is less pronounced for concretes with higher MWP. It is observed that the final loss reaches 49% for the control concrete, whereas it is only 35.6% for concrete incorporating 18% MWP.



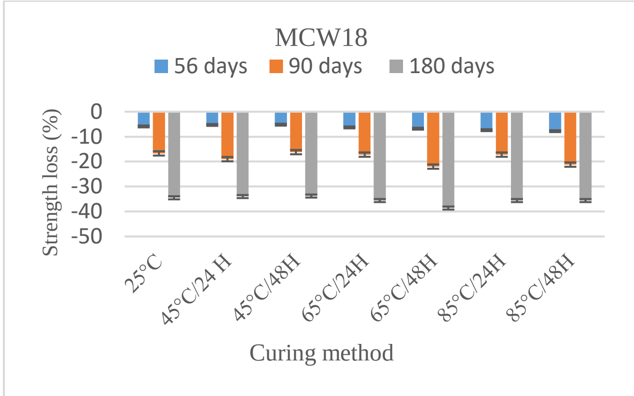
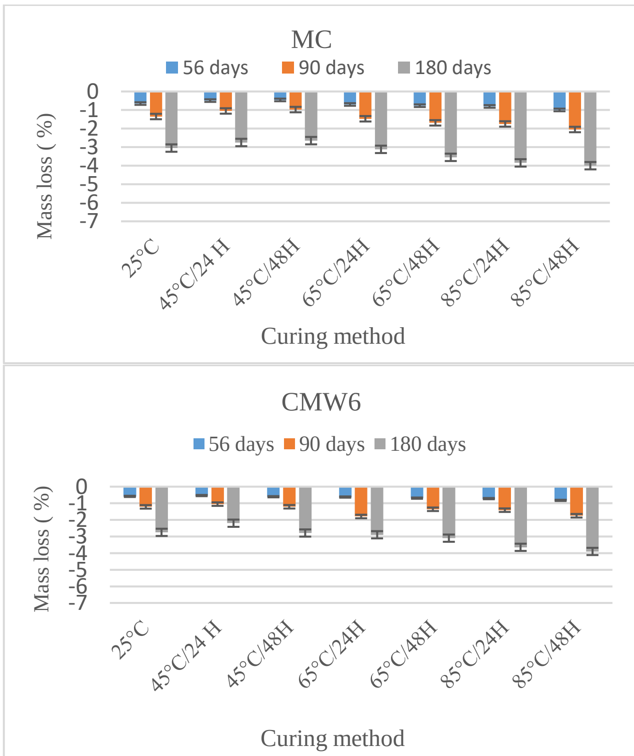


Figure 5. Strength loss (%) of concrete mixes after hydrochloric acid attack (HCl)

4.4.2 Mass loss

According to Figure 6, MWP exhibits a positive effect on the resistance against HCl ions. Increasing MWP content results in a significant reduction in the final mass loss. A final mass loss by 0.65% is observed for the control concrete, whereas this loss is reduced to 0.54% for concrete containing 18% MWP. This trend was also shown by Gameiro et al. [43] and Vardhan et al. [44]. It can be also noted that excepting the control concrete cured at 45℃, which shows a decrease of 0.40% in final mass loss, mass loss increases with increasing of the curing temperature. The final mass loss at 25℃ is 3.05%, whereas it reaches 4% at a temperature of 85℃ for the control concrete mixture. The increase in curing time notably affects the final mass loss. The maximum loss value for a 24-hour curing period is 3.85%, whereas for a 48-hour curing period, it reaches 4% for the control concrete mixture cured at 85℃. This increase in mass loss for concrete thermally treated at higher temperature is attributed to the increase in water absorption and porosity of concrete.



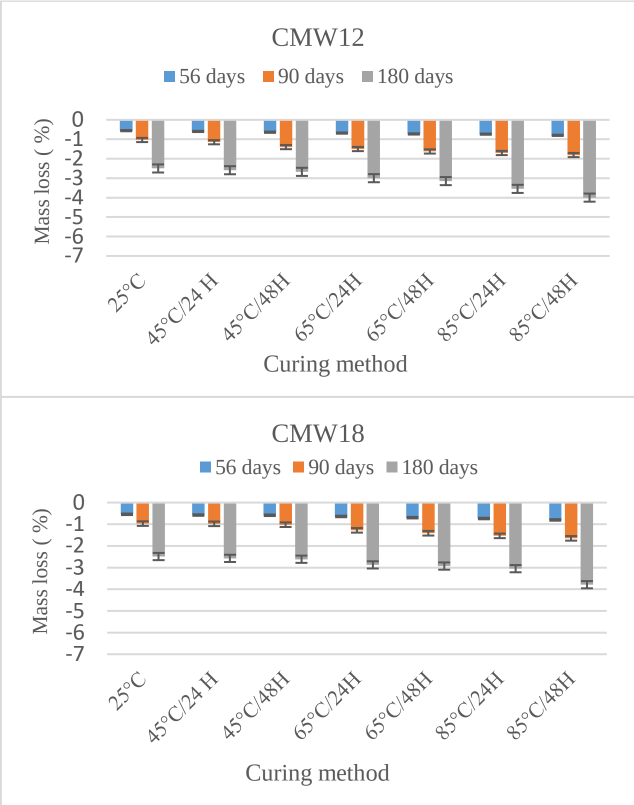
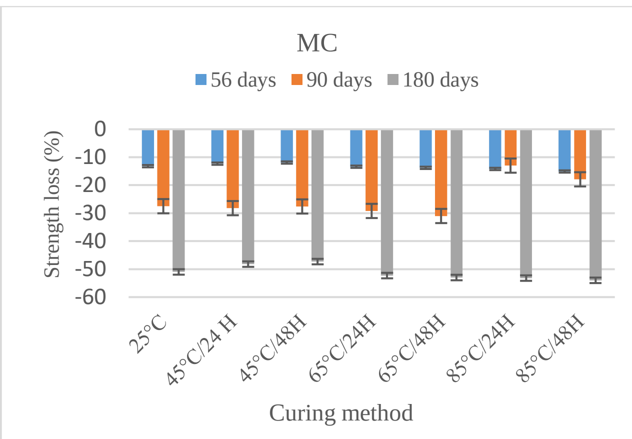


Figure 6. Mass loss of concrete mixes after hydrochloric acid attack (HCl)

4.5 Chemical attack by H_2SO_4

4.5.1 Compressive strength loss

Figure 7 shows that concretes immersed in H_2SO_4 solution exhibit similar behavior to that immersed in HCl solution. In fact, the incorporation of MWP reduces the final resistance loss to H_2SO_4 ions compared to the control concrete at 25°C. Specifically, the concrete containing 18% MWP exhibits a loss by 41%, while the CM mixture shows a loss by 51%. Additionally, there is a 7.9% reduction in resistance loss for concrete containing 18% MWP and treated at 45°C, compared to the CM mixture. However, when the temperature exceeds 45°C, the resistance loss becomes more pronounced, reaching 54% at 85°C for CM mixture. As the compressive strength loss, the addition of MWP mitigates the negative effect of thermal treatment, leading to a 10.7% reduction in final loss for the concrete containing 18% MWP at 85°C.



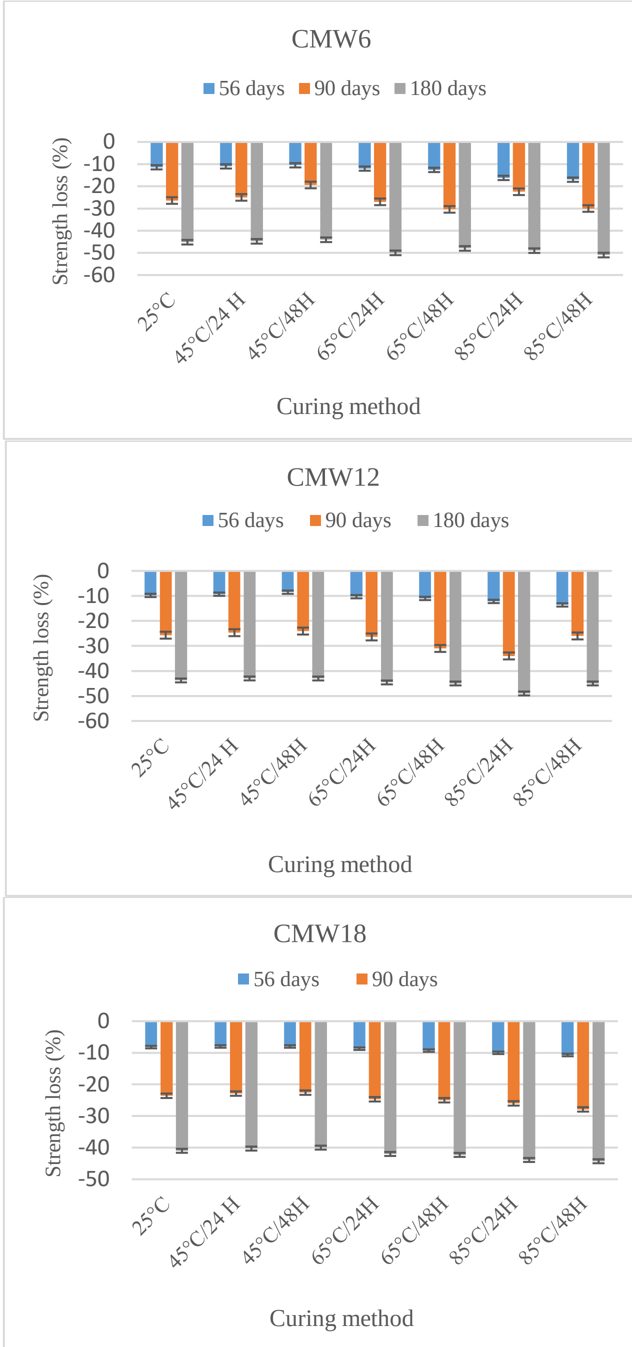


Figure 7. Strength loss (%) of concrete mixes after sulfuric acid attack (H_2SO_4)

4.5.2 Mass loss

According to Figure 8, increasing the MWP content to 18% leads to a reduction in the final mass loss, which decreases from 0.79 to 0.66% for the control concrete.

The thermal treatment at 45°C has a positive effect on the final mass loss of concrete, resulting in a loss by 3.8%, while the loss is 4.4% at 25°C. When the temperature exceeds 45°C, the loss becomes more significant, reaching 6.55% at 85°C. Furthermore, the incorporation of MWP helps to overcome the negative effect of the increase in the curing temperature. At 85°C, the final mass loss of concrete modified with 18%MWP is 4.56%, while for the control concrete the loss in mass is 6.55%. These results agree with that of water absorption which increases with the increase in temperature treatment, facilitating the penetration of ions.

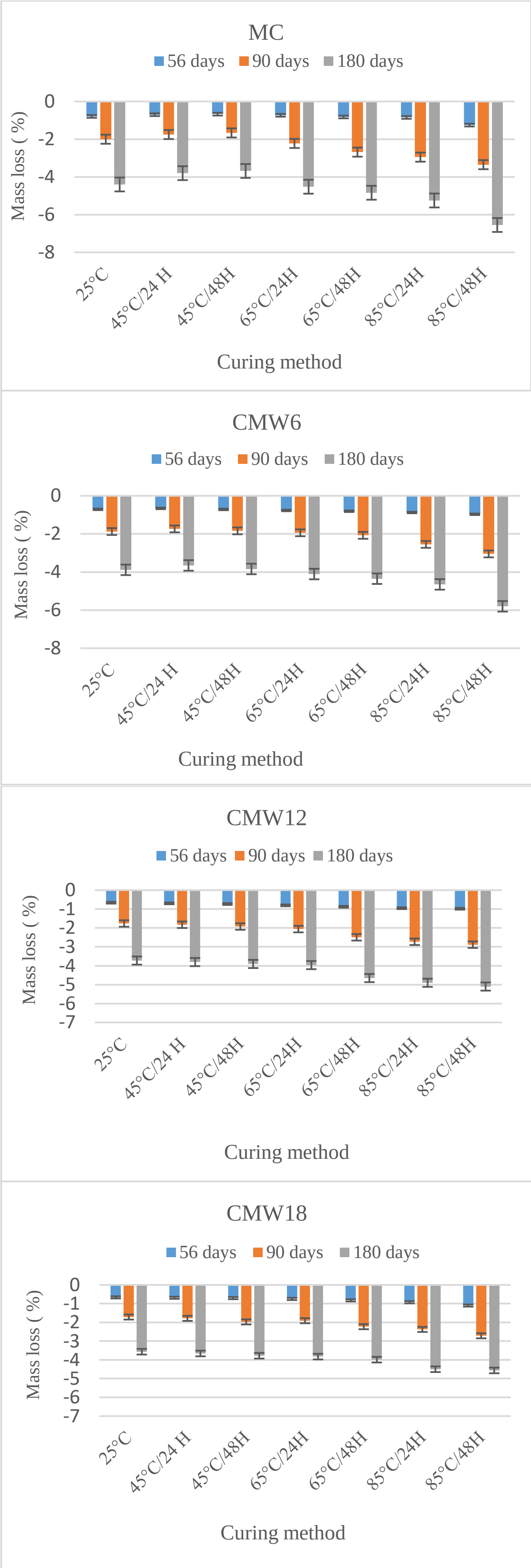


Figure 8. Mass loss (%) of concrete mixes after sulfuric acid attack (H_2SO_4)

5. CONCLUSIONS

In this study, the effect of temperature treatment on mechanical and durability properties of concrete modified with 6%, 12%, and 18% MWP were examined. Based on the experimental results obtained in this work, the following conclusions can be drawn:

-The increase in the temperature positively affects the early compressive strength which increases by 60.28% at 2 days for concrete modified with 18% MWP and treated at 45℃. However, the long-term strength of thermally treated concrete tends to decrease by 29.92 for concrete without MWP. The increase in curing duration also decreases the mechanical properties of concrete. It is noteworthy that thermal treatment at 45℃ for 24 hours represents the optimal curing method.

-The incorporation of MWP in concrete composition has beneficial effect on mechanical properties of concrete thermally treated. In fact, the presence of MWP reduces the decline in compressive strength of thermally treated concrete over the long term. With 18% of MWP, the reduction in compressive strength of concrete cured at 85℃ for 48 hours decreases from 29.92% to 1.64%.

-Thermal treatment enhances the flexural strength of concrete at early age which increased by 54.84% at 2 days for CMW12 mixture treated at 45℃. It is important to highlight that curing at 45℃ for 24 hours represents the optimal method.

- Thermal treatment has negative effect on the UPV values of concrete containing MWP. The UPV generally decrease with increasing of the curing temperature. This decrease in the UPV is accompanied by an increase in water absorption by 0.99% of concrete modified with MWP

-Thermal treatment at 45℃ is found to improve the concrete's resistance to HCl and H_2SO_4 ions, while higher temperature treatment decreases the resistance of concrete. The incorporation of MWP decreases the strength loss by 13.40% and 10.70% for concrete immersed in HCl and H_2SO_4 solutions, respectively.

Finally, it can be noted that the introduction of MWP as a cement replacement has a positive effect on most mechanical and durability properties of concrete thermally treated. In addition to reducing the negative effect of higher curing temperatures on concrete strengths, MWP improves concrete resistance to the chemical attacks. To obtain the optimum properties, it is recommended to use a heat treatment regime of 45℃ for 24 hours with a MWP content that should not exceed 12%.

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