Effects of plasticizer and antifreeze on concrete at elevated temperatures and different cooling regimes

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Abstract
In this study, the plasticizer and the antifreeze were used. The concrete specimens (the reference concrete without an admixture, concrete with plasticizer, concrete with antifreeze and concrete with plasticizer+antifreeze) were exposed to elevated temperatures (200, 400, 550 and 700 ºC) and cooling via air and water. Water absorption, ultrasonic pulse velocity and compressive strength tests were performed on the specimens. The concrete specimens were also analyzed using thermal gravimetric analysis and X-ray diffraction. The test results indicated that at the temperatures of 550 and 700 ºC and against both of the cooling regimes the plasticizer+antifreeze concrete showed a maximum strength loss. When the air cooled specimens were examined, the lowest strength loss was this obtained for reference concrete at 550 and 700 ºC. The lowest strength loss at these temperatures for the water cooled specimens was observed in the concrete with antifreeze. According to thermal gravimetric analysis, the weight loss rate slightly decreases after 700 °C. No portlandite peaks were observed in the concrete mixtures exposed to 700 ºC and the cooling regimes was when they were compared at with the control specimens exposed to 20 ºC in the X-ray diffraction analysis.

Keywords: concrete, plasticizer, antifreeze, elevated temperatures, cooling regimes

Introduction

Fire poses a serious threat to the safety of structures. Structures can be both directly and indirectly exposed to high temperatures. The fire resistance parameter should not be ignored during the design of buildings. The choice of building materials has a very important role in the fire resistance of a building as the failure to demolish a building during a fire depends on the durability of the construction materials of the building. The most widely used material in the construction of structures all over the world is concrete. In this context, it is important to know the usual behavior of concrete and the behavior it displays during fires. The studies on the behavior of concrete against high temperatures are still ongoing (Papachristoforou et al., 2020; Mehdipour et al., 2020). When concrete is exposed to high temperatures, chemical changes occur in its structure, its physical properties become damaged and it losses strength. Thus, the effects of aggregate size and type, cement type and dosage, mineral admixtures, nano and fibrous materials on concrete at elevated temperatures and different cooling regimes have been widely investigated by several researchers (Karahan et al., 2019; Alharbi et al., 2020; Gökçe, 2019; Afzal & Khushnood, 2020, Silva et al., 2020).

Chemical admixtures have become one of the main components of concrete alongside aggregate, cement and water. As an important part of concrete, different types of chemical admixtures have been widely used to improve its properties, such as good workability and high durability (Tong et al., 2020). One of the most commonly used admixtures is water reducing plasticizer. Plasticizers are admixtures that allow for the reduction of water in concrete without affecting its consistency or increase the slump without affecting the water content (TS EN 934-2, 2011). Antifreeze, on the other hand enables concrete to gain strength in cold weather by reducing the freezing point of the cement paste liquid phase and accelerating the hydration of the cement (Polat, 2016). The use of antifreeze is economical and common to reduce the negative impact of cold weather conditions on concrete (Khan & Kumar, 2020). Concretes containing chemical admixtures can exhibit different behaviors compared to those without chemical admixtures when they are exposed to high temperatures (Maanser et al., 2018). Ahmad and Abdulkareem (2010) determined that admixtures can be considered as one of the factors that affect the heat-treatment of concrete with other factors. There are very few studies on the effects of chemical admixtures at elevated temperatures. Some researches regarding the topic are summarized below:

Aruntas et al. (2009) investigated the effect of superplasticizers on concrete at high temperatures. In their study, the reference concrete and concrete with superplasticizer were exposed to 20, 100, 200, 300, 400, 500 and 700 ºC. They determined that the compressive strength of the concrete without an admixture decreased by 50% at 400 °C and 72% at 700 °C, while the compressive strength of the concrete with the superplasticizer decreased by 42% at 400 °C and 78% at 700 °C.

In another study conducted by Ahmad and Abdulkareem (2010), concrete mixtures containing super plasticizer, plasticizer, retarding and water reducing admixture, accelerating admixture, air entrainers were exposed to 200, 400 and 600 ºC. They observed that the highest residual compressive strength at 200 °C was obtained from the concrete with plasticizer. The highest residual compressive strength 400 and 600 °C, on the other hand, were obtained from the concrete containing the retarding and water reducing admixture.

Regardless of the chemical admixture type, concrete are exposed to high temperature effects during fire. According to the literature review, there was no study conducted on the strengths of concrete containing antifreeze after being exposed to high temperatures and different cooling regimes. Thus, in this study, plasticizer and antifreeze were preferred and were used to prepare four concrete mixtures. The influence of different chemical admixtures on the development of the high temperature strength with cooling regimes was studied and compared.

Experimental program

Materials

CEM I 42.5 R Portland cement conforming to EN 197-1 (2012) standard was used for all of the mixtures in the study. The cement's specific fineness was 3320 cm^2/g. Water conforming to EN 1008 (2013) requirements was used in the concrete mixtures. The water was potable and did not contain any harmful substances. The maximum grain size of the limestone aggregates was 16 mm. The specific gravities of aggregates are 2.72 for 0-4 and 4-8 mm; 2.74 for 8-16 mm. Water absorption results of the aggregates are 1.43%, 0.96% and 0.74% (TS EN 1097-6, 2013).

Two types of well-known commercial admixtures, namely inorganic salt-based antifreeze and lignin sulfonate-based plasticizer, were used in this study (ASTM C494, 2016; TS EN 934-2, 2011). The pH value of the antifreeze was 6.00 and its solution density was 1250 kg/m^3. The pH value of plasticizer, on the other hand, was 6.50 and its solution density was 1060 kg/m^3.

Methods

The concrete mixture for the experimental investigations were prepared according to the TS 802 (2016) and TS EN 206-1 (2017) standards. The concrete class of the specimens was C30/37. The plasticizer and the antifreeze were used at 1% rate of the cement weight. The admixtures were added into the mixing water. The mixture proportions for 1 m^3 concrete are given in Table 1. In this study, four concrete mixtures were produced. The mixtures were coded as ref for the concrete without an admixture, P for the concrete with plasticizer, A for the concrete with antifreeze and PA for the concrete with plasticizer+antifreeze. In concrete mixtures, W/C are different in order to keep the slump values equal.

Table 1. The mixture proportions for 1 m^3 concrete.

Concrete mixtures	W/C ratio	Materials						
		Cement (kg)	Water (l)	Plasticizer (l)	Antifreeze (l)	Aggregate (kg)		
						0–4 mm	4–8 mm	8–16 mm
Ref	0.60	350	210	0	0	908	544	368
P	0.55	350	193	3.5	0	922	552	370
A	0.59	350	207	0	3.5	917	550	370
PA	0.53	350	187.5	3.5	3.5	939	564	380

Firstly, cement and both fine and coarse aggregate were placed in a laboratory mixer and mixed for 1.5–2 min. Then the admixtures were added into water. After the water was added to the mix, all of materials were mixed for 3–3.5 min. (TS EN 12390-2, 2010). Slump test was carried out in fresh concrete (TS EN 12350-2, 2010). The average value of the slump was 11 cm for the concrete mixtures. Fresh concrete both with and without the admixtures were cast into 15x50x60 cm plate molds (TS 1247, 2018). Three molds were used for each concrete type. The cylindrical core specimens were extracted from these molds on the 14^{th} day (TS EN 12504-1, 2011). In this study, total 600 core specimens were taken from the concrete slabs to obtain a better representation of the material. The top and bottom of the core specimens were cut. The diameter of the cylinder samples was 50 mm and the height was 100 mm. They were kept in the water cure pool until the 90^{th} day at 20 ± 2 °C.

After 90 days of water cure, concrete specimens were dried at 100± 5 °C for 24 h in an oven to remove the excess water. 150 specimens of each concrete mixture were used. Then the concrete specimens were thermally treated in an oven at 200, 400, 550 and 700 °C for 3 hours to evaluate the effect of the elevated temperatures, 30 specimens of each temperature were used. Then, half of the specimens were cooled at an ambient temperature (20 ºC) while the other half were cooled with water.

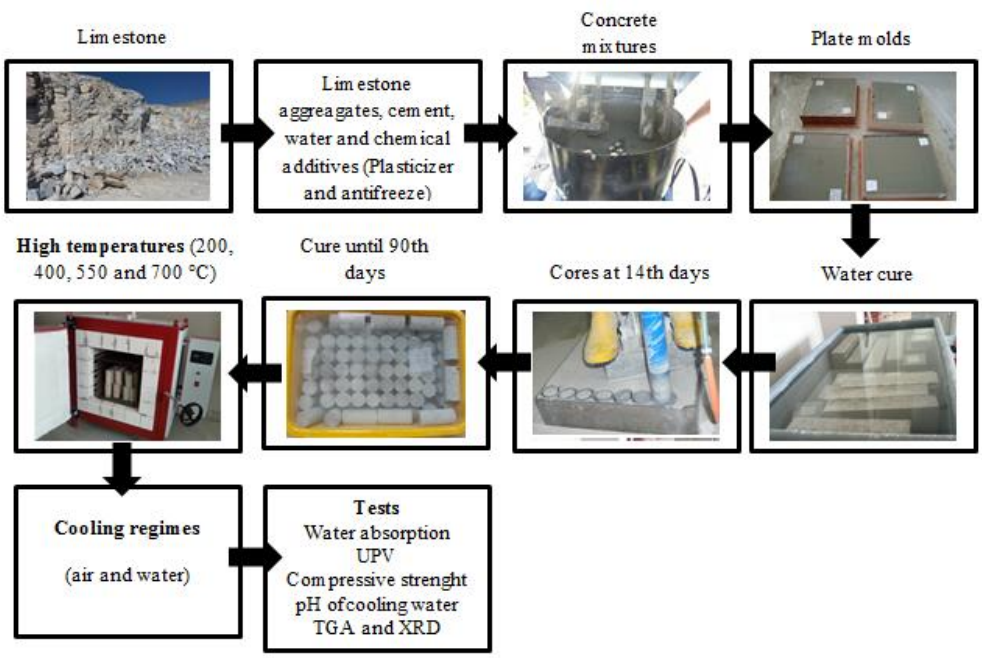
Water absorption was performed according to ASTM C642 (2004) principles, ultrasonic pulse velocity (UPV) according to ASTM C 597-83 (2016) principles and compressive strength tests according to EN 12390-3 (2010) principles on Ø50x100 mm cylindrical concrete specimens after the heating and cooling processes. Test results were compared with the control specimens which were stored in laboratory conditions after drying at 100±5 °C.

The thermal analysis test is a technique that determines the change in the weight of the sample due to the temperature increase. It was performed with a PERKIN ELMER STA-600 device. A 15 mg weight is placed on the thermonanobalance of the apparatus and weighted accurately throughout the test. The powder samples were heated from 20 °C to 900 °C with a 20 °C per minute heating rate. The thermogravimetric curve (TGA) and the derived thermogravimetric curve (DTG) were obtained on four concrete type.

XRD allows the qualitative and quantitative chemical identification of the crystalline phases found in the material at high temperatures (Caetano et al., 2019). Core specimens stored at 20 ºC and exposed 700 ºC were crushed and milled for standard qualitative mineral analysis by XRD method. The diffraction patterns of the samples were obtained at room temperature, in the X-pert Pro model powder diffractometer system of Panalytical brand and by using copper radiation [λ(CuKα) = 1.54056Å] between 2° and 70° theta screening range.

The methods used in the study are shown in Figure 1.

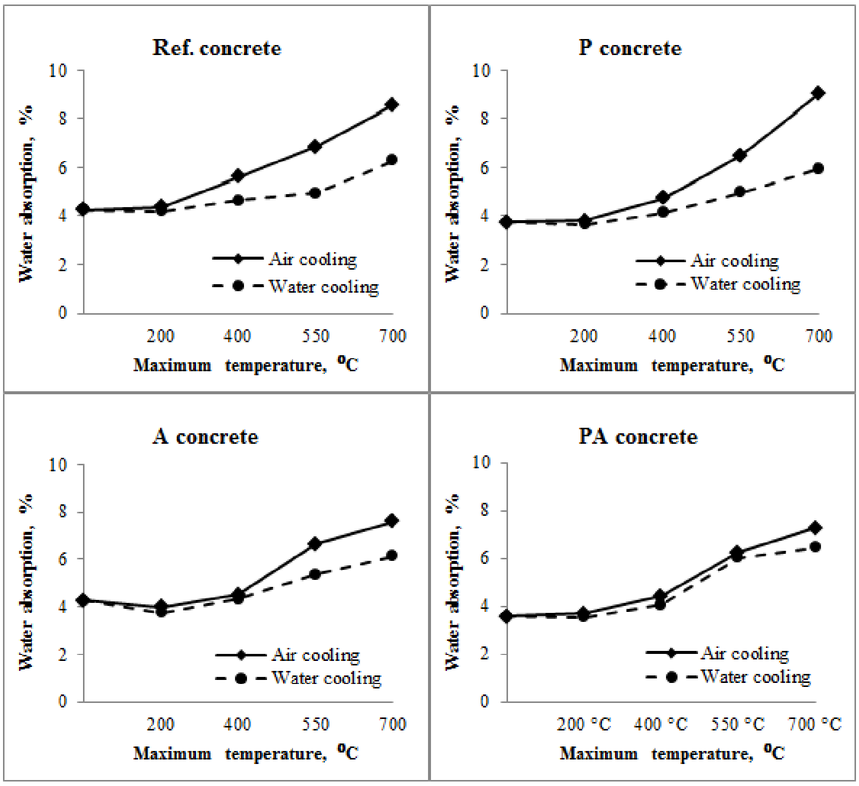
Figure 1.The methods.



Water absorption capacities

The 90th day water absorption results of the concrete mixtures exposed to the heating and cooling regimes are exhibited in Figure 1. Figure 1 displays an overview of the effect of the elevated temperatures on the water absorption capacities of the concrete mixtures compared to the control mixture, which was exposed to 20 °C. After being heated to 200 ºC and then cooled via water cooling the water absorption properties of the ref, P and PA mixtures decreased. The most significant reduction was observed in the P mixture. The water absorption capacity of the water cooled P specimens decreased from 4.78% to 3.76% at 200 °C. Water absorption was significantly influenced by the cooling regimes at 550 and 700 °C for the ref, P and A mixtures. The cooling regime did not create a large difference in the PA mixture. The water absorption capacity of concrete generally increases with the increase in temperature. It is also known that pores and micro cracks in concrete increase as a result of high temperature exposure. This has a great impact on the water absorption capacity of concrete (Hiremath and Yaragal, 2018). The water absorption capacities of the air-cooled specimens were greater than the water cooled specimens for all of the mixtures. In this context, it can be said that air cooling causes more pores and micro cracks.

Figure 2. Water absorption results of all mixtures exposed to the heating and cooling regimes. Source: Self-Elaboration.



In Table 2, the differences in the water absorption capacities of the concrete mixtures exposed to elevated temperatures and cooling regimes can be seen. When the temperature reached 550 and 700 ºC, the water absorption capacities of the air cooled samples increased rapidly. It is thought that this may cause the occurrence of excessive voids due to the dehydration of C-S-H gel (Salahuddin et al., 2019).

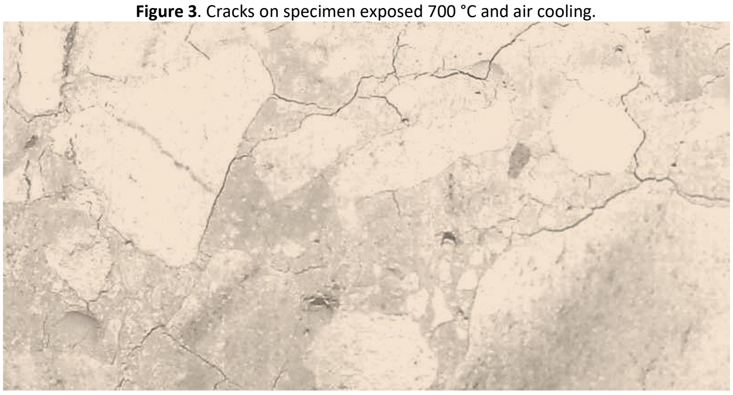
Table 2. The effect of high temperature and the cooling regimes on the difference of water absorption (↓: decrease, ↑:increase). Source: Self-Elaboration.

Difference of Water adsorption, %									
Temperature, °C		200	400	550	700	200	400	550	700
Cooling regime		Air cooling				Water cooling			
Concrete type	Ref	↓0.81	↑15.68	↑33.83	↑56.35	↓2.33	↑5.10	↑5.44	↑25.54
	P	↑1.04	↑19.47	↑35.28	↑72.00	↑2.14	↑7.37	↑12.24	↑27.71
	A	↓4.77	↑3.88	↑32.69	↑45.33	↓7.15	↑0.41	↑14.84	↑25.55
	PA	↑4.92	↑16.95	↑43.18	↑54.55	↑2.20	↑16.17	↑25.74	↑38.13

UPV

UPV test is a qualitative test used to evaluate the quality of concrete. It is a technique that is sensitive to degradation phenomena including internal cracking and other deterioration due to thermal treatment (Hiremath and Yaragal, 2018). The UPV of the specimens were measured on the 90th day. The minimum UPV value was obtained from the A concrete mixture as 3.56, while the maximum UPV value was obtained for the PA concrete mixture as 3.78 km/s. The UPV test was carried out once again after the specimens were exposed to the high temperatures and the cooling regimes. However, the UPV values of the specimens subjected to the air cooling regime could not be measured at 550 and 700 °C. As is known, ultrasonic gel is applied to the surfaces where the receiver and transmitter tips are applied in the measurement of UPV. Therefore, with this gel, a curtain against air is formed between the ends and the concrete surface. These specimens have cracks on the surface (Figure 3). The UPV device did not measure due to mutual air passage or the formation of gaps, due to cracks around the cylinder. Therefore, the results for the specimens exposed 550 and 700 °C and air cooling are missing in Figure 4. The results for the UPV test conducted on the 90th day for the concrete mixtures exposed to the high temperatures and cooling regimes are exhibited in Figure 4.

Figure 3. Cracks on specimen exposed 700 °C and air cooling.



At 200 ºC, the results for the air cooled specimens varied between 3.39 and 3.66 km/s, while the results for the water cooled specimens varied between 3.27-3.46 km/s. At 400 ºC, these results ranged between 1.77-2.33 km/s and 1.92-2.19 km/s for the air and water cooled specimens, respectively. At 550 and 700 ºC, the results varied between 1.32 and 1.48 km/s for the air cooled specimens and 1.00-1.25 km/s for for the water cooled specimens. It is known that there is a relationship between mechanical properties and UPV results as UPV results depend on pore structure and specimen density (Benaicha et al., 2015). It was thought that the pores of the air cooled samples were more than those of the water cooled specimens at 550 and 700 ºC.

Figure 4. UPV results of all of the mixture exposed to the high temperatures and cooling regimes. Source: Self-Elaboration.

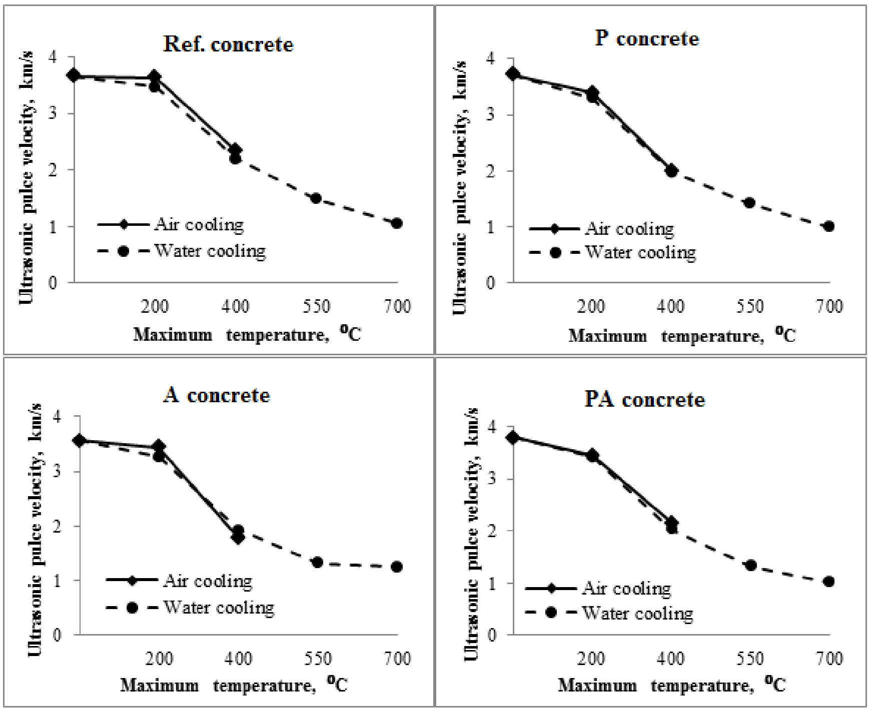


Table 4 shows the decrease in the UPV values after the exposure to the high temperatures and the cooling regimes. As the temperature increased, the UPV values of the air cooled ref, P, A and PA concrete mixtures reduced by 0.82%, 8.87%, 3.37% and 8.73% at 200 °C, respectively. These results increased to 36.34%, 46.24%, 50.28% and 46.30% km/s for 400 ºC, respectively

It was determined that the water cooled specimens showed a higher decrease in UPV values at 200 and 400 °C compared to the air cooled specimens. After being heated to 550 ºC, the UPV values of the water-cooled ref., P, A and PA concrete mixtures reduced by 59.56%, 61.83%, 62.92% and 65.08%, respectively. It is known that micro cracks in the cement paste and aggregates begin to spread between 450-600 ºC. Thus, the UPV results showed a decrease of more than 50% (AzariJafari et al., 2019). The minimum UPV decrease at 700°C was observed in the water cooled A concrete mixture as 64.89%, while the maximum UPV decrease was observed in the water cooled PA concrete mixture as 73.28% at 700 ºC.

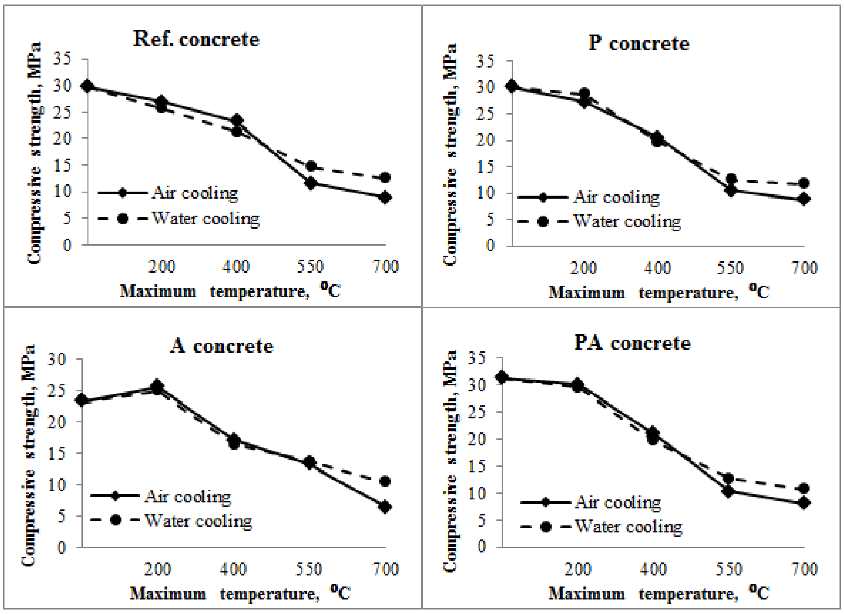
Table 4. Decreases in the UPV values of the concrete specimens after being exposed to the high temperatures and cooling regimes (↓: decrease). Source: Self-Elaboration.

Difference of UPV, %									
Temperature, °C		200	400	550	700	200	400	550	700
Cooling regime		Air cooling				Water cooling			
Concrete type	Ref	↓0.82	↓36.34	↓100.00	↓100.00	↓5.46	↓40.16	↓59.56	↓71.31
	P	↓8.87	↓46.24	↓100.00	↓100.00	↓11.29	↓47.04	↓61.83	↓73.12
	A	↓3.37	↓50.28	↓100.00	↓100.00	↓8.15	↓46.07	↓62.92	↓64.89
	PA	↓8.73	↓46.30	↓100.00	↓100.00	↓9.52	↓46.30	↓65.08	↓73.28

Compressive strength

The compressive strength values of the concrete specimens after being exposed to the high temperatures and cooling regimes are shown in Figure 5. The compressive strengths of all of the concrete mixtures reduced rapidly after being exposed to 400 ºC. A further reduction in the compressive strengths of the water cooled specimens was observed at 200 and 400 °C compared to the air cooled ones. This finding was in parallel with those in the literature. Wang et al. (2019) reported that strength deterioration was more severe for concrete samples subjected to water cooling compared to those subjected to natural cooling, especially at the temperatures of 200 and 400 °C. However, the opposite of this situation occurred as the temperatures increased. The air cooled specimens were more damaged than the water cooled ones at 550 and 700 ºC. The reason for this is that cement mortar decomposes at high temperatures, however, when water cooling is carried out, the components are re-hydrated. The air cooled specimens were found to be denser than the water cooled specimens. It is known that cooling regimes have an effect on residual compressive strength (Luo et al., 2010). Durgun and Sevinc (2019) reported that air cooled specimens were more exposed to atmospheric conditions. In such cases, CaO produced by $CaCO_3$ decomposition is recycled and volume expansion occurs. According to Karakoç (2013), in actual situations fires in buildings are extinguished using water. However, there are very few studies that have been conducted on cooling regimes.

Figure 5. Compressive strength results of all of the mixtures exposed to the high temperatures and cooling regimes. Source: Self-Elaboration.



The specimens of each concrete type that had been kept at 20 ºC were accepted as the control specimens and their compressive strengths were accepted as 100%. The differences in the compressive strengths of the specimens were calculated by using the control strengths and the strengths obtained after the specimens were exposed to the high temperatures (Table 5).

Table 5. The effect of the high temperatures on the compressive strength (↓: decrease, ↑:increase). Source: Self-Elaboration.

Difference of strenght, %									
Temperature, °C		200	400	550	700	200	400	550	700
Cooling regime		Air cooling				Water cooling			
Concrete type	Ref	↓9.13	↓21.71	↓60.94	↓70.21	↓13.18	↓28.24	↓50.62	↓57.80
	P	↓9.47	↓31.85	↓64.93	↓71.07	↓4.78	↓34.77	↓58.45	↓61.04
	A	↑9.61	↓26.94	↓42.74	↓72.50	↑6.62	↓29.97	↓41.72	↓55.51
	PA	↓3.54	↓32.84	↓66.92	↓74.41	↓5.39	↓37.02	↓59.69	↓65.74

At 200 °C, the compressive strengths of the ref, P and PA mixtures decreased in both the air and water cooled specimens. However, the compressive strength of the A concrete mixture increased in both cooling conditions. The increase in compressive strength between the temperature range of 105-200 °C can be explained by the further hydration of dehydrated cement particles and/or an improvement of the local mechanical properties of C-S-H (Lim and Mondal, 2015). Previous studies determined that the cause of this phenomenon is the thermal expansion stress generated in concrete due to heating (Hwang et al., 2018).

Air cooling at 400 ºC caused the compressive strength of the specimens to decrease in the range of 21.71-32.84%, while this range was 28.24-37.02% for water cooling at the same temperature. The compressive strengths of the air cooled specimens exposed to 550 ºC decreased in the ranges of 42.74–66.92%. This range was between 41.72% and 59.69% for the water cooled specimens at the same temperature. It is known that when concrete is exposed to a temperature above 400 ºC, the $Ca(OH)_2$ in the concrete begins to dehydrate and compressive strength decreases by 45% (Dexing et al., 2018). At 700 ºC, the decrease in compressive strength was higher for the air cooled specimens compared to the water cooled specimens. The decrease in compressive strength of the air cooled specimens exceeded 70.00% for all of the mixtures. The decreases in the compressive strength of the water-cooled specimens, on the other hand, were in the ranges of 55.51–65.74% at the same temperature. It is known that after 550 °C, as almost all C-S-H gel and $Ca(OH)_2$ crystals decompose and the internal structure of concrete is further damaged and cracked, the compressive strength further decreases (Wang et al., 2017).

Properties of the cooling water

The following were observed when the pH changes of the cooling water after high temperature application were examined (Table 6):

- Increased cooling water pH with the increase in temperature.
- The pH value of the cooling water of PA at 700 ° C was 8.62, however this value changed to 10.22 after the cooling process.

It is thought that the increase in the pH value according to the increase in temperature occurred due to some of the CaO dissolving in water and converting into $Ca\ (OH)_2$.

Table 6. pH values of the cooling water

Temperature, ºC	pH of water	Concrete mixtures			
		Ref	P	A	PA
200	Before cooling	8.08	8.12	8.30	8.45
	After cooling	8.14	8.26	8.42	8.66
400	Before cooling	8.24	8.28	8.26	8.45
	After cooling	8.38	8.42	8.41	8.68
550	Before cooling	8.41	8.29	8.37	8.54
	After cooling	8.48	8.39	8.65	8.88
700	Before cooling	8.42	8.47	8.53	8.62
	After cooling	9.42	9.46	9.44	10.22

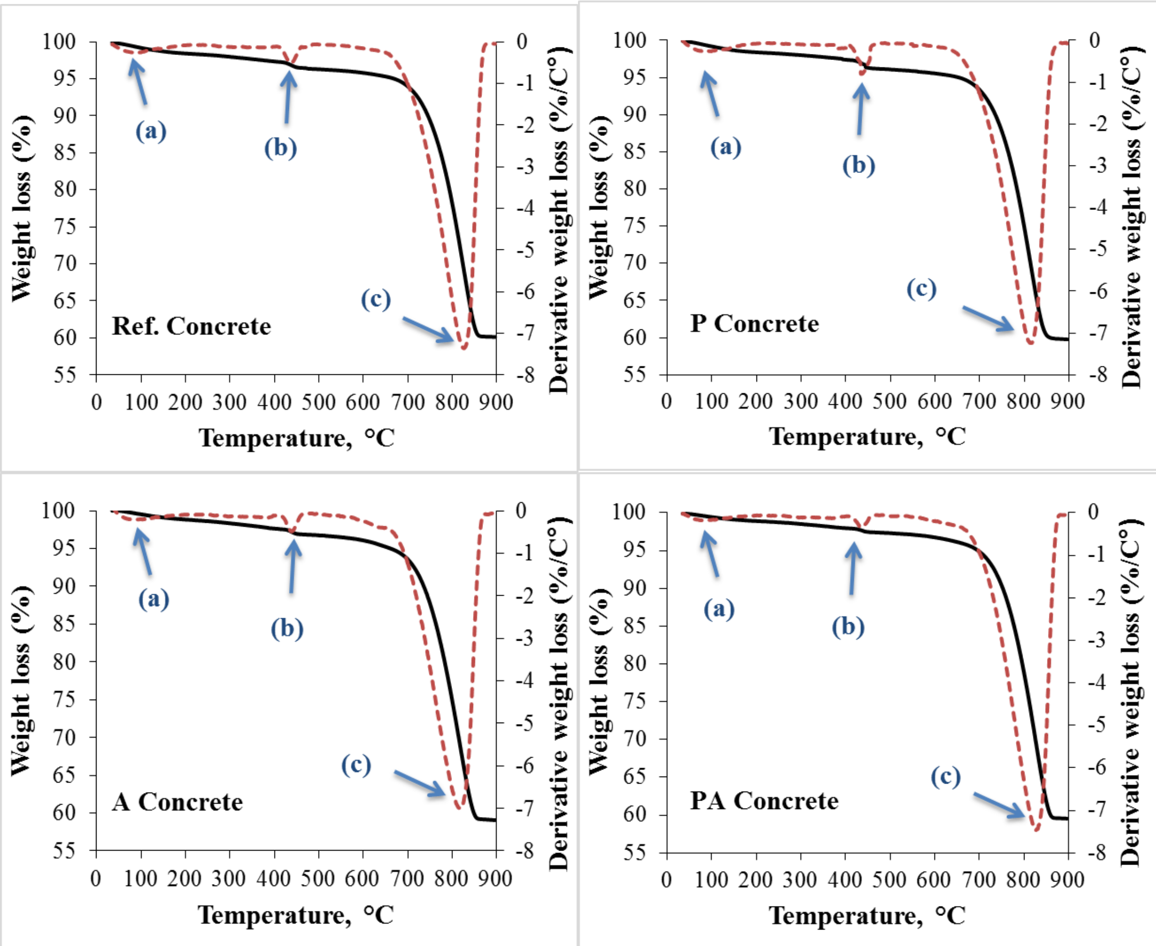
The pH of the cooling water changed the most in the PA concrete mixture. This was in parallel with the decrease in compressive strength. The concrete mixture that lost the most compressive strength at 400, 550 and 700 °C the PA concrete mixture. It was determined that the concrete type most prone to being damaged from temperature and cooling conditions had the cooling water with the highest pH difference.

Thermo gravimetric analysis

TGA/DTG curves are presented in Figure 6. The weight loss at 200 °C is 1.59%, 1.61%, 1.16% and 1.60% for Ref., P, A and PA concrete, respectively. The weight loss at 400 °C is 2.66%, 2.61%, 2.36% and 2.03% for Ref., P, A and PA concrete, respectively. The weight loss at 550 °C is 3.90%, 4.13%, 3.50% and 2.89% for Ref., P, A and PA concrete, respectively. The weight loss at 700 °C is 6.01%, 6.67%, 6.42% and 5.72% for Ref., P, A and PA concrete, respectively. The weight loss rate slightly decreases after 700 °C. The decomposition process starts at 760 °C and finishes abruptly at 846 °C for Ref. concrete. It starts at 761 °C and finishes 831°C for P concrete. It starts at 768 °C and finishes 831 °C for A concrete. It starts at 790 °C and finishes 850 °C for PA concrete.

Total weight loss is about 39%, 40%, 41% and 42% for Ref., P, A and PA concrete, respectively. The peaks result from water evaporation (a), decomposition of portlandite at 400-500 °C (b) and decomposition of calcium carbonate between 700 and 850 °C (c) in Figure 6.

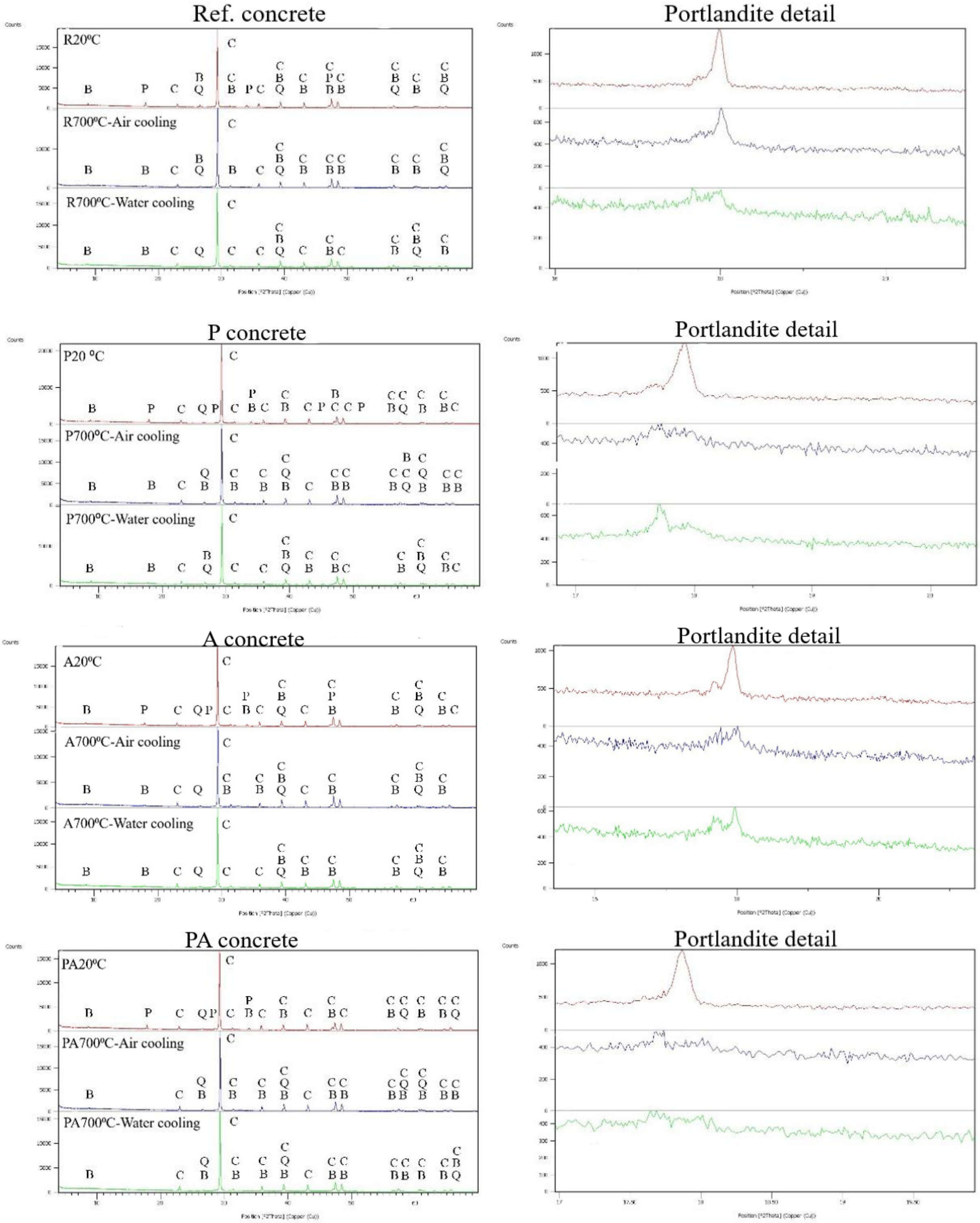
Figure 6. TGA/DTG curves of concrete mixtures. Source: Self-Elaboration.



X-ray diffraction analysis

XRD analysis was used to determine the changes in the mineralogical structures of the concrete types after being exposed to 700 ºC and the cooling regimes. Figure 7 shows the diffraction patterns of the concrete mixtures.

Figure 7. XRD pattern of the detached concrete pieces from the specimens (P: Portlandite, C: Calcite, Q: Quartz) and the portlandite detail. Source: Self-Elaboration.



It can be seen from Figure 6 that there were more quartz and calcite peaks in all of the mixtures than portlandite peaks. A portlandite peak was seen at 2θ = 18° in the ref. specimens exposed to 20 ºC. However, after the exposure to 700 ºC and the cooling regimes, the XRD analysis showed no diffraction peak for portlandite. According to the XRD results, there was no difference between the concrete mixtures.

Conclusions

In this study, the compressive strengths of concrete specimens containing chemical admixtures were determined after being exposed to high temperatures and cooling regimes, namely air cooling and water cooling. The experimental tests were carried out on four concrete mixtures: ref mixture (concrete without an admixture), P mixture (concrete with plasticizer), A mixture (concrete with antifreeze) and PA mixture (concrete with plasticizer+antifreeze). Within the scope of this study water absorption, UPV and compressive strength tests were conducted at the temperatures of 20, 200, 400, 550, 700 °C. The results obtained in this study are as follows:

- The highest compressive strength was obtained for the PA concrete mixture at 20 ºC. The water absorption and UPV test results of this mixture were also in support of the compressive strength results.
- The water absorption categories of all of the concrete mixtures increased with the increase in temperature above 200 ºC for both cooling regimes. According to the differences in the water absorption capacities, the air cooled specimens were more damaged than the water cooled ones.
- When the cooling regimes were compared it was determined that the air cooled specimens at 400 ºC had less damage compared to the water cooled ones. On the other hand, when temperatures reached 550 and 700 ºC, the water cooled specimens showed better resistance.
- The concrete type that suffered the least damage at 550 and 700 °C was the water cooled A concrete mixture. The compressive strength decrease of the A concrete mixture was 55.51% at the maximum temperature.
- The concrete type most damaged at 550 and 700 ºC was the air cooled PA concrete mixture. A 74.41% decrease was observed in the strength of this mixture at the maximum temperature.
- When the pH of cooling water was examined, it was found that the highest pH difference was for the PA concrete mixture at all temperatures.
- Total weight loss is about 39%, 40%, 41% and 42% for Ref., P, A and PA concrete, respectively according to TGA.
- XRD patterns for all of the concrete mixtures were similar. After being exposed to 700 ºC and the cooling regimes, the XRD analysis showed no diffraction peak for portlandite.

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