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Influence of curing regimes on compressive strength of ultra high performance concrete

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Abstract. The present paper is aimed to identify an efficient curing regime for ultra high performance concrete (UHPC), to achieve a target compressive strength more than 150 MPa, using indigenous materials. The thermal regime plays a vital role due to the limited fineness of ingredients and low water/binder ratio. By activation of the reaction kinetics, the effectiveness of the binder is enhanced which leads to improvements in mechanical as well as durability properties. The curing cycle employed are ambient air curing, water curing and hot air curing. The specimens were exposed to thermal regime at (90°C/150°C/200°C) for duration of 24, 48 or 72 hours at the age of 3rd and 7th day followed with air curing or water curing till 28 days. The results showed a marked difference in compressive strength ranging from 217 to 142 MPa with change in curing regimes. The samples when thermally cured at the age of 3rd and 7th day produced an average ultimate strength of 217–152 MPa and 196–150 MPa, respectively.

Keywords. Ultra high performance concrete (UHPC); curing cycle; hot air curing; water curing; air curing; compressive strength; thermal regime.

1. Introduction

The bridges and highways are being constructed inlarge number all around the globe using UHPC, there is need to study the performance and behaviour of UHPC using Indian materials. The properties of UHPC are different from high performance concrete and normal concrete. The curing methodology plays a major role for full development of microstructural attributes. The Association Française de Génie Civil (AFGC) in its interim recommendations states UHPC to have the following properties: Compressive strength that is greater than 150 MPa, internal fibre reinforcement to ensure non-brittle behaviour, and high binder content with special aggregates. The constituents are cement, fine sand, silica fume, quartz powder, superplasticizer, a low water-cement ratio, and inclusion of either high-strength steel fibres or non-metallic fibres

(AFGC 2002). The basic principles for developing UHPC are optimization of granular mixture, elimination of coarse aggregate, microstructure enhancement by heat curing, addition of steel fibres for ductility and densification of cementitious matrix through vibration, post/pre pressurizing the concrete (Richard & Cheyrezy 1995).

UHPC is branded with different names all around the globe such as Ductal, Core TUFF, BSI, UHPFRC (Ultra High Performance Fibre Reinforced concrete), Reactive Powder Concrete (RPC), UHSFRC (Ultra-high strength fibre reinforced concrete). It has compressive strengths more than seven times and tensile strength exceeding three times that of ordinary concrete (Graybeal & Hartmann 2003). The main reason for the high functionality of UHPC is the active participation of all materials in pozzolanic reaction. It has been observed that neither the physical and chemical structure of the hydration products is changed by the hydration temperature up to 45°C. The reduction in the pore structure plays a vital role for high mechanical and physical performance. At heat treatments of 200°C the samples show a residual expansion after return to normal atmospheric temperature, which is due to the expansion of C–S–H gel with tobermoritic characteristic that, fills the vacant space. For temperature above 200–250°C the porosity and threshold pore size are increased which are due to formation of xonotlite accompanied with formation of water. A minimum value of porosity, corresponding to very small pore diameter is obtained for pressed samples with heat treatments between 150°C and 200°C (Richard & Cheyrezy 1995; Cheyrezy *et al* 1995). Higher temperature induces microstructural changes of the hydrate structure; it alters and accelerates the pozzolanic reaction of the silica fume and quartz. The tricalcium silicates and dicalcium silicates present in the cement lead to the formation of calcium silicate hydrate ($C_3S_2H_3$) popularly known as C–S–H gel. At high temperature and pressure it converts itself into a crystalline product known as α- dicalcium silicate hydrate ($\alpha - C_2S$-H) which is detrimental strength wise, as it is enriched with higher porosity, density and smaller volume of solid phase. On continued heating of around 200°C, CaO/SiO_2 ratio reduces and C–S–H ultimately converts to xonotlite (C_6S_6H) and smaller content of tobermorite ($C_5S_6H_5$) and scawtite ($C_7S_6CH_2$) which is having a very dense micro structure (Richard & Cheyrezy (1995), Cheyrezy *et al* (1995), Feylessoufi *et al* (1997), Redaa *et al* (1999), Kamen *et al* (2007)). The stoichiometric equations involve in the hydration of the cement are as follows-

$$2\,C_3S + 6H \rightarrow C_3S_2H_3 + 3CH \tag{1}$$

$$2\,C_2S + 4H \rightarrow C_3S_2H_3 + CH \tag{2}$$

$$C_3S_2H_3 \rightarrow C_6S_6H + C_5S_6H_5 + C_7S_6CH_2 \tag{3}$$

$$^{*}C - CaO, S - SiO_2H - H_2O$$

Water curing (WC), hot air curing (HAC), steam curing (SC) are the most popular methods employed to provide necessary environment for the concrete to inhibit hydration which depends on the type of cement, admixture and water binder ratio. The compressive strength of UHPC samples is dependent on the hydration of cement, pozzolanic reactions of silica fume at lower temperatures and combined action of silica fume and quartz at higher temperatures. Due to slower rate of hydration it has become inevitable to induce thermal regime to initialize the reactions. Hence proper thermal regime is required to cause proper development of micro-structure of CSH hydrates. by the heat treatment (Cheyrezy *et al* 1995). The properties of UHPC vary with the difference in temperature curing and duration. At 20°C, 26% degree of hydration is reached after 90 days. A linear relationship is observed between the degree of hydration,

compressive strength and modulus of elasticity. The curing temperature is responsible for the thermo-activation of the hydration reaction which effects the initial and final setting time. The elevated temperature extrapolates autogenous shrinkage which is due to self-desiccation and the capillary depression influenced by low water/binder ratio (Kamen *et al* 2007). The controlled rate thermal analysis (CRTA) shows the formation of xonotlite at a temperature range below 200°C and on higher temperatures truscottite, gyrolite and hillebrandite are formed depending on the CaO/SiO_2 ratio (Feylessoufi *et al* 1997) XRD investigations on UHPC using calcined bauxite, silica fume and silica flour has denser micro structure with improved mechanical performance having weak CH crystals to strong C–S–H gel (Redaa *et al* 1999). XRD investigations also revealed no formation of tobermorite crystals at 90°C and 160°C (Lee & Chisholm 2005). The compressive strength results showed that auto clave curing is superior to steam curing. The accelerated hydration process leads to 25–63 % and 9–61 % gain in strength by steam curing and autoclaving compared to standard curing. The steam curing stimulates the constituents while, autoclaving leads to microstructural development at different phases stating that addition of silica is vital to attain higher mechanical properties (Halit *et al* 2009). A comparative study between 0.16 and 0.20 binder ratio shows 200 % increment in the strength after steam and dry heating curing (Juanhong & Shaomin 2011). The experimental results revealed that UHPC specimens cured at 20°C performance was 20 %, 10 % and 15 % lower to cured samples at 90°C with respect to compressive strength, flexural strength and fracture energy, respectively (Yang *et al* 2009).

2. Present investigations

2.1 *Mix design*

The mix design of UHPC at CSIR-SERC was based on the reference mixes available in the literature and various trial and errors done at the laboratory. It is observed that silica fume/cement ratio and quartz powder/cement is 0.25 and 0.40, respectively. The cost-effective optimal dosage of steel fibres is equivalent to a ratio of 2% by volume, or about 155 kg/m^3. Sand is used here as a filler and superplasticizer is added in the mix to provide workability in the mix. Table 1 presents various mix proportions for UHPC obtained from available literature to get the most compacted and highest density. Richard & Cheyrezy (1995), Barnett *et al* (2007), Harish *et al* (2011, 2008), Prabhat *et al* (2012). The density of mixture obtained was in the range of (2460–2520) kg/m^3.

Table 1. Comparison of UHPC mixes in literature and present study.

Composition	Richard and Cheyrezy (1995)	Ductal (Dallaire *et al* (1998)	Dili & Santhanam (2004)	Graybeal (2007)	Present Study
Cement, kg/m^3	865.1	705	718.4	710	788
Silica-fume, kg/m^3	199	230	179.6	230	197
Quartz, kg/m^3	337.4	210	222.7	210	315
Fine aggregate, kg/m^3	951.6	1010	1199	1020	866.8
Water, l/m^3	147.1	195	179.6	110	173
SP, l/m^3	20.21	17	26.93	31	14.77
Steel fibre, kg/m^3	–	190	–	156	157
w/c ratio	0.17	0.27	0.25	0.16	0.22

Table 2. Chemical composition of cement and silica fume.

Oxides	Sio_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	Mn_2O_3	SO_3	Free Lime	Chlorides	LOI
OPC	20.49	5.91	4.07	62.90	1.13	0.20	0.47	0.20	0.08	1.87	0.45	0.012	2.29
SF	94.73	–	–	–	–	0.51	–	–	–	0.2	–	0.07	1.5

2.2 *Materials properties*

2.2a *Ordinary Portland cement*: The cement used during the experiments is ordinary Portland cement of grade 53 conforming to IS 12269:1987. According to IS 4031, the tested 28-day mortar compressive strength is 58 MPa. The specific gravity is 3.15; the initial and final setting times are 110 min and 260 min. The normal consistency being 28% and the particle size range lies between 31 μm and 7.5 μm. The chemical composition of cement is given in table 2.

2.2b *Silica fume*: The silica fume used in the experiment conforms to ASTM C1240 – 97b. The specific gravity being 2.25, percentage passing through 45 μm sieve in wet sieve analysis is 92 % and the particle size range lies between 5.3 μm and 1.8 μm. The chemical composition of silica fume is given in table 2.

2.2c *Quartz powder*: The specific gravity being 2.59, percentage passing through 45 μm sieve in wet sieve analysis is 75 % and the particle size range lies between 5.3 μm and 1.3 μm.

2.2d *Sand*: The sand used for the experimental studies are Grade I -Coarse (particle size range of 0.6 mm–2.36 mm) and Grade III Fine (particle size range of 0.075–0.15 mm).

2.2e *Super plasticizer*: Poly-acrylic ester-based type SP was used.

2.2f *Steel fibres*: Micro-steel fibres- having an aspect ratio of 81 was chosen with length of 13 mm, diameter 0.16 mm and tensile strength of 2000 MPa. The volume fraction of 2% was used for the mix.

3. Experimental investigations

For initial trials, Mix A and Mix B were considered. The basic differences in the two mixes were the fillers quartz sand and ennore sand respectively. The mix proportion is given in table 3. The trail mixes were then exposed to curing cycles P, Q and R. The sets were repeated for hot air curing at temperature of 90°C and 150°C, to find out the appropriate temperature for hydration of the ingredients. An ultra thermal cyclic chamber model was used for hot air curing. The temperature range for this varies till +300°C, ±1.0°C. The rate of rise in temperature is 2°C/min. The details of the curing regime cycle are as follows. Cycle P – The samples were cast and after demoulding kept in water for curing till the date of testing (28th day).

Table 3. Mix proportion of mix A and mix B.

Materials	C	SF	Q	QC	QF	ES-1	ES-3	W/C	SP	SF
Mix A	1	0.25	0.4	0.71	0.30	–	–	0.23	1.5%	2%
Mix B	1	0.25	0.4	–	–	0.71	0.30	0.23	1.5%	2%

SF – Silica Fume, C – Cement, Q – Quartz Powder , QC- Quartz coarse and QF- Quartz fine, ES – Ennore Sand, W – Water, SP – Super Plasticizers, SF- Steel Fibres

Table 4. Compressive strength (MPa) at the end of 28 days for Mix A and Mix B.

Type of curing	Cycle P*	Cycle Q*		Cycle R*	
Temperature	25°C	90°C	150°C	90°C	150°C
	Compressive strength (MPa)				
Mix A	82	87	113	110	131
Mix B	101	110	137	122	152

Table 5. Mix proportion for Mix C.

Materials	C	SF	Q	ES-1	ES-3	W/C	SP	SF
Mix C	1	.25	0.4	0.71	0.30	0.20	1.5%	2%

Table 6. Compressive strength (MPa) at the end of 28 days for Mix C.

Type of curing	Cycle P	Cycle Q
Temperature	25°C	200°C
	Compressive strength (MPa)	
Mix C	140	171

Cycle Q -The cubes were casted, demoulded and after setting in ambient air curing, of laboratory environment it is subjected to thermal regime having temperature of 90°C and 150°C for 72 hours at the age of 3rd day. After thermal curing, the samples were taken out of the oven and after they attain thermal equilibrium with atmospheric temperature they were water cured till the date of testing (28th day).

Cycle R - The procedure was same as the Cycle Q, the only major difference was temperature curing being done on the 7th day to see the effect of delayed thermal curing. The result of compressive strength at the age of 28 days for Mix A and Mix B is given in the table 4. Based on the preliminary studies, it was observed that Mix B containing ennore sand as filler produced higher compressive strength than Mix A. A better mix proportion Mix C as shown in table 5 with ennore sand as filler and w/c ratio of 0.20 was tried then with higher temperature of 200°C. The samples were then cured with cycle P and Cycle Q at 200°C. The Mix C produced better results with compressive strength more than 150 MPa as shown in table 6. Hence for detailed investigation, Mix C was used to identify an optimal curing regime.

For detailed investigations 13 curing cycles were repeated. The descriptions of them are as follows: CR1- The specimens after demoulding are kept in water till the age of 28 days.

CR2, CR3, CR4 specimens after demoulding are kept in water and then exposed to hot air curing at 200°C for the duration of 24, 48 and 72 hours from the 3rd day, after which they were allowed to attain thermal equilibrium and then kept in water till the age of 28 days. The procedure is same for CR5, CR6, CR7 as CR2, CR3, CR4 but instead of keeping the samples in water after the thermal curing they are continued for air curing till the date of testing. CR8, CR9, CR10 specimens after demoulding are kept in water and then exposed to hot air curing at 200°C for the duration of 24, 48 and 72 hours from the 7th day, after which they were allowed to attain thermal equilibrium with the atmosphere and then kept in water till the age of 28 days. CR11, CR12, CR13 specimens procedure is same as CR8, CR9, CR10 but instead of keeping the samples in water after the thermal curing they are continued for air curing till the date of testing. The measured temperature of water is 25°C and air is 30°C.

3.1 *Mixing*

A Planetary mixer machine (15 kg capacity) was used to mix the UHPC. The speed can be varied from stir (slow), speed (low), speed 2 (medium), speed 3 (high) having agitator RPM of 59, 107, 198 and 365, respectively. The dry binder powder was poured in the bowl and dry mixing was done for 3 minutes each at slow and low speed. Around 70% of the water and super plasticizer was added and the level of mixing was increased to medium, which was continued for another 3 min. When proper blending of ingredient was observed, the remaining SP and water were added and mixed at high speed for 5 min again. After mixing it is observed that the materials have gained proper fluidity and homogeneity. The level of mixing is then changed to slow speed where a random distribution of fibres was done taking approximately 3 min. Finally, at high speed the ingredients were fully mixed for 2 min to have a flowable consistent mix.

4. Results and discussions

(i) Test for compressive strength was carried out on 70 mm cube specimens under water saturated surface dry condition in a 3000 KN capacity Compressive Testing Machine (CTM). Test arrangement and method used for compression tests were in accordance with IS: 516–1959. The values were calculated by taking average of four samples for a particular reading.

(ii) It is observed that ennore sand acts better filler than quartz powder from the compressive strength results obtained for Mix A and Mix B as shown in table 5. Thermal regime of 200°C and w/c ratio of 0.20 gave more compressive strength than thermal regime at 90°C and 150°C.

(iii) Elaborate trials were done to find out the optimum duration for the hot air curing by using Mix C. The compressive strength of the specimens tested at different age of the concrete with varying curing regime is plotted in figures 1 to 4. It was observed the all the UHPC samples when cured in water (CR1) showed significant increase in the strength with the curing period. The compressive strength obtained at the end of 28 days is 142 MPa. But the case was not the same when cured at higher temperature. When the UHPC samples are exposed to thermal regime, the variation in compressive strength is very wide from 217 to 150 MPa. The results as shown in figure 1 show CR3 is the best regime. It is also observed that 72 hours of hot air curing is not required to cure the specimens while 24 hours of hot air curing leads to 16% decline in the compressive strength. The samples with CR 5–7, as shown in figure 2 had ultimate strength in the range of 172 – 152 MPa. The CR 8–CR 10

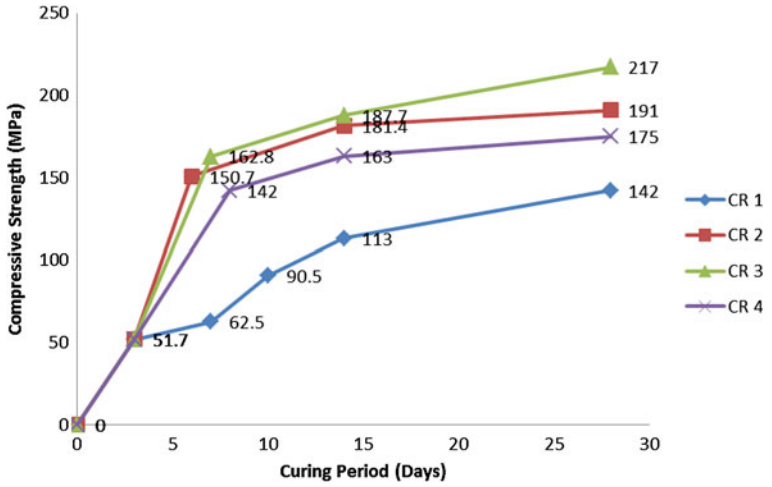

Figure 1. Compressive strength (MPa) for CR1, CR2, CR3 and CR4 versus curing period (days).

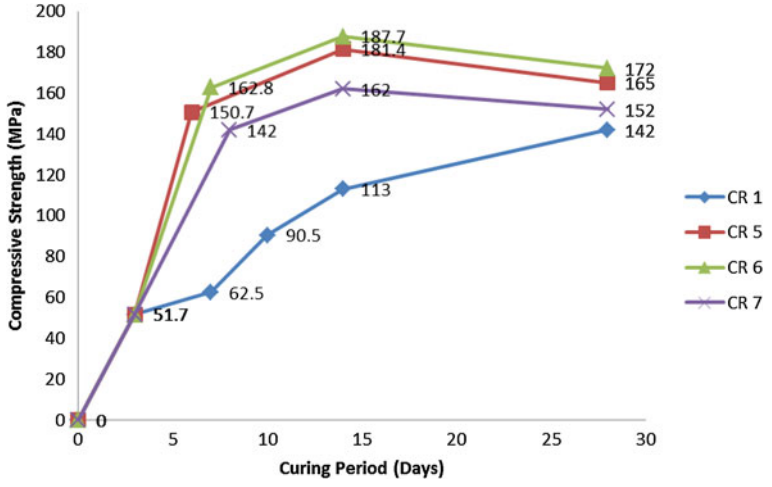

Figure 2. Compressive Strength (MPa) for CR1, CR5, CR6 and CR7 versus curing period (days).

as shown in figure 3 attain almost same compressive strength in the range of 196–190 MPa after 28 days. Hence 24 hours of thermal regime is sufficient to obtain strength of 193 MPa. Compressive strength in the range of 175–150 MPa was obtained from CR 11–CR 13.

(iv) The thermal regime is very important to activate silica fume and quartz powder otherwise the material will act as filler and not as a binder. The hot air cured samples showed dramatically rise in the compressive strength but there was a decrease in strength for the samples which were thermally cured for longer duration. The possible reason for the behaviour could be high early age autogenous shrinkage as well as rapid surface drying and surface cracking because of its low water–binder ratio and addition of high fineness admixtures (Yoo *et al* 2013). The presence of silica fume results in higher creep strain (Igarashi *et al* 2000). The

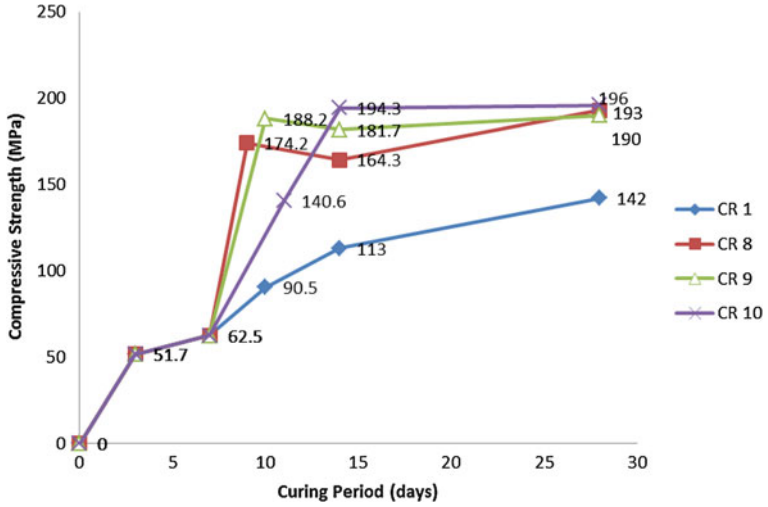

Figure 3. Compressive Strength (MPa) for CR1, CR8, CR9 and CR10 vs curing period (days).

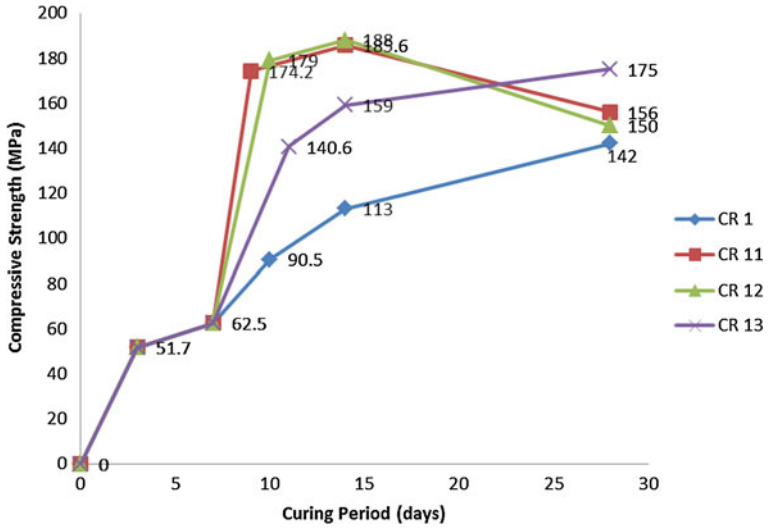

Figure 4. Compressive Strength (MPa) for CR1, CR11, CR12 and CR13 vs curing period (days).

chemical reactions of binders develop simultaneously and in an interdependent way, leading to solid skeleton progressive formation and hydration products rearrangement, which are responsible for strength development. The hydration progress and the strength development are strongly temperature dependent. The effect of temperature can be attributed to two effects: (i) a kinetic effect, that is, increase of temperature results in the activation of hydration and the evolution of the strength and the decrease of temperature slows down the hydration process; and (ii) a thermodynamic effect, that is, decrease of the strength at long term and rearrangement of hydrates under high temperatures (Kamen *et al* 2007, 2008).

Verbeck & Helmuth (1969) suggested that curing at higher temperatures resulted in a non-uniform distribution of the hydration product within the microstructure, with a high concentration of hydration product building up around hydrating grains and retarding subsequent hydration, while at low temperatures hydration products have sufficient time to diffuse and precipitate relatively more uniformly throughout the cement matrix. UHPC is subjected to thermal regime in order to provide accelerated curing and hence enhance the development of mechanical properties, so that it can widely be used for industrial applications like precast elements, repair and rehabilitation in less time (Gowripalan & Gilbert 2000; Lee & Chisholm 2005; Cwirzen 2007).

(v) The activity of the admixtures mainly comes in to action when they react with calcium silicate hydrates, calcium hydroxide and aluminate hydrates. Defloccution and dispersion of the cement particles occur due to negative charge attained by them resulting in repulsion among them. There are possible two ways of exploitation of this phenomenon either to increase the workability or enhance strength. In the present study, the SP was used to restrict the w/c ratio to 0.20, in order to gain high strength. For the same amount of SP the procedure for addition of SP was very vital to produce better workability. For trial mixes it was observed that the step-wise addition of the SP produced better flowability of the concrete. For few trials the whole SP and water was added separately, in some of them SP mixed with water was added, while step-wise addition of 70% of (SP with water) after dry mixing and remaining at some interval produced best results. Poly-acrylic ester type super plasticizers were used for the study. (vi) The results of curing cycles can be used by industry to obtain desired compressive strength ranging from 213–142 MPa using Indian materials.

5. Conclusions

(i) The compressive strength results showed that ennore sand constituting (71% of Grade I and 30% of Grade II can act as better filler than quartz sand.

(ii) The samples when thermally cured after 2 days with hot air curing for 24, 48 and 72 h at (200°C), continued with water curing or air curing produced an average ultimate strength of (213–153) MPa. The delayed thermal regime at the age of 7th day produced an average ultimate strength of (196–155) MPa. This clearly depicts that a proper curing cycle is very important to obtain the best results.

(iii) The rate of hydration and strength gain for UHPC is very high till the age of 14 days. The samples gain 85% of the compressive strength at the age of 14 days. From this study a curing cycle of 48 h of hot air curing at 200°C after demoulding followed with water curing is suggested, to gain compressive strength of more than 210 MPa, using indigenous materials. When the samples are not given temperature regime and only water cured a 65 % decrease in ultimate strength is observed compared to suggested cycle as above, which clearly signifies the importance of proper curing regime in the study.

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