Influence of Initial Water Curing on Strength and Microstructure Development of Blended Cements

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Abstract. Hydration of cement is a long term chemical process which requires water. Construction industry often prefers cement with high early strength and short curing period. Limestone calcined clay cement (LC3), which can replace clinker up to 50%, has potential benefits in this regard due to its low clinker factor and rapid reaction kinetics. In this study, ordinary portland cement, fly ash based portland pozzolanic cement, limestone calcined clay cement and composite cement (OPC with slag and fly ash) were prepared in laboratory. Mortar cubes and paste specimens were cast at constant water to binder ratio. The samples then exposed to different curing regimes by changing it from water curing to air curing (R.H $\approx$ 10–20%) at selected intervals. The curing temperature was kept constant as 27 °C. Compressive strength of mortar cubes were taken at standard ages. Effect of curing conditions on phase assemblage were quantified using, quantitative x-ray diffraction.

1 Introduction

Hydration of cement requires water. The process of curing ensures the presence of water in the system during hydration process, by providing moisture externally or stopping evaporation of water from specimen. Generally, the rate of hydration is high during the initial days and then decreases, making the initial days critical from curing point of view. But, this depends on type of cement. The industry is keen to reduce the use of clinker in cement, by replacing it with alternative materials called supplementary cementitious materials (SCMs). The cement blends with lower clinker factor have many advantages over conventional ordinary Portland cement (OPC). These blends can drastically reduce environmental pollution that comes from production of clinker through lowering the clinker factor by utilizing industrial by-products such as fly ash and slag. As per the Indian standards, based on quality, up to 30% of clinker can be replaced by fly ash and, up to 70% of clinker can be replaced using slag. Generally, these binary blends have low early strength and longer curing period [1]. The construction industry is interested in high early strength as well as lower curing period due to economic factors. In order to reduce these disadvantages, ternary cements were proposed using more than one SCMs [2].

The curing requirement may be different for different type of cements. It is expected that cements with high early age strength demands high amount of water. Ternary cements using alternative raw materials may influence the curing requirements. Many studies have

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shown that the effect of curing conditions is more influential on blended cements. A study on fly ash based cement indicated that effect of curing largely depends on physical and chemical properties of clinker and w/b ratio rather than that of fly ash properties and replacement factor [3].

This study aims to identify the effect of curing conditions: relative humidity and curing duration on strength development of mortar from different blends prepared by replacing clinker with various SCMS.

2 Materials and Methods

2.1 Raw Materials and Blends

The raw materials were ground individually in laboratory ball mill. Blends were prepared by interblending of raw materials. The chemical composition of raw materials from X-ray fluorescence and composition of raw materials in blends are shown below (Tables 1 and 2).

Table 1. Chemical composition of raw materials

Chemical composition (%)	Clinker	Gypsum	Fly ash	Slag	Cal. Clay	Limestone
SiO_2	21.07	2.77	67.66	32.26	54.67	11.02
Al_2O_3	4.65	0.62	22.18	23.16	27.69	1.55
Fe_2O_3	4.32	0.36	5.32	1.93	4.93	2.53
CaO	65.16	32.62	0.32	33.88	0.06	44.24
MgO	2.13	1.2	0.18	7.01	0.13	1.96
SO_3	0.77	38.75	0.02	0.00	0.10	0.00
Na_2O	0.38	0.06	0.43	0.01	0.12	0.00
K_2O	0.20	0.037	1.6	0.37	0.25	0.00
LOI	0.96	23.02	1.74	1.08	10.28	36.96

Table 2. Raw material composition of blends (by % of weight)

Name of the blend	Abbr.	Clinker	Gypsum	Fly ash	Slag	Calcined clay	Lime-stone
Ordinary portland cement	OPC	95	5	–	–	–	–
Portland pozzolanic cement	FA30	65	5	30	–	–	–
Composite cement	CCS	50	5	15	30	–	–
Limestone calcined clay cement	LC3	50	5	–	–	30	15

2.2 Specimen Preparation

For compressive strength testing, mortar cubes of 70.6 mm size were cast as per IS 4031 part-6. The blends were dry mixed with standard sand [4] with a ratio of 1:3. A fixed water to binder ratio-0.45 was used for all blends. Paste samples were prepared by mixing cement blend with water at w/b ratio of 0.45. Proper mixing was ensured using high speed paste mixer.

2.2.1 Curing Conditions

The temperature during the whole process: material conditioning, casting of specimen and curing was maintained at 27 ± 2 °C. The prepared specimens (mortar cubes and paste samples) kept in an environmental controlled room (temperature: 27 ± 2 °C, RH: 65 ± 5%) for 24 h. After first day, specimens were demoulded and shifted to different curing regimes. Two different relative humidity (RH) was considered for this study: A higher RH of 100% (HH) and a lower RH of 10–20% (LH). HH was obtained by placing samples in lime saturated water and LH was obtained by placing samples in a closed box with silica gel inside. Fresh silica gel, which is blue in colour absorbs moisture from surroundings and turns colourless. Old silica gel was continuously replaced by fresh one at regular intervals to keep the humidity low. The curing duration was planned by shifting specimens from HH to LH at specific time durations. The details of curing durations and corresponding notation used in this paper are shown in Table 3.

Table 3. Curing regimes and notations

Curing regime	Notations
Specimen cured in HH	HH
Specimen cured in HH till 3 day and shifted to LH after 3 days	HH(3)_LH
Specimen cured in HH till 7 day and shifted to LH after 7 days	HH(7)_LH
Specimen cured in LH	LH

The cubes were tested in compression testing machine of capacity 500 kN with a loading rate of 2.40 kN/s. Average compressive strength of 3 cubes was considered for representation. The test is performed as per the Indian standard [5]. The compressive strength tests were carried out on the specimens in mortar cubes at 28 days.

For x-ray diffraction analysis, hardened Cement paste slices of 3 mm were cut and one surface of the slice was polished using 1200 silicon carbide paper. The XRD scanning was carried out in Bruker D8 Advance Eco X-ray diffractometer at 40 mV and 25 mA with Cu target (K_α = 1.54 A^o). The range of scanning is 5^o to 65^o and rate of scanning is 0.019^o/0.3 s. The whole running time for a single sample is around 17 min. In order to avoid carbonation of samples, scanning was carried out immediately after slicing and polishing. Quantitative analysis of phases was carried out using the software TOPAS v5 from Bruker. Major clinker phases and hydration products were quantified by external standard method using rutile.

3 Results

The 28-day compressive strength of mortar cubes cured under various curing regimes are shown in Fig. 1 (Fig. 2).

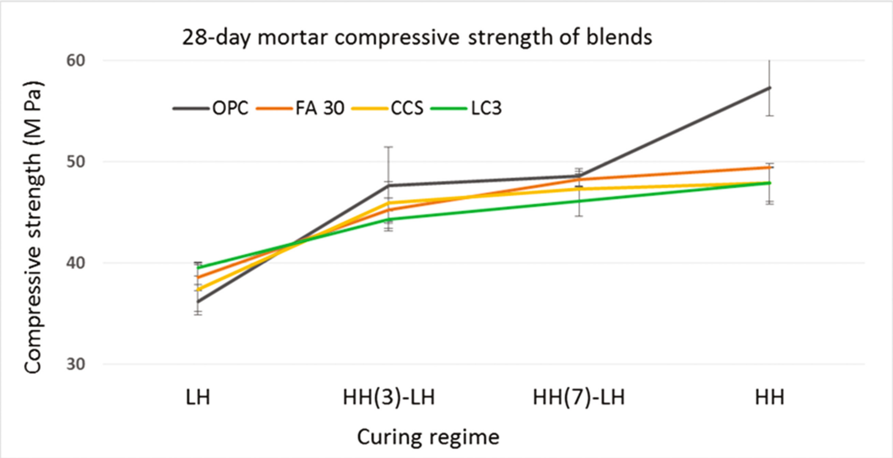

Fig. 1. 28-day mortar compressive strength of blends at different curing regimes

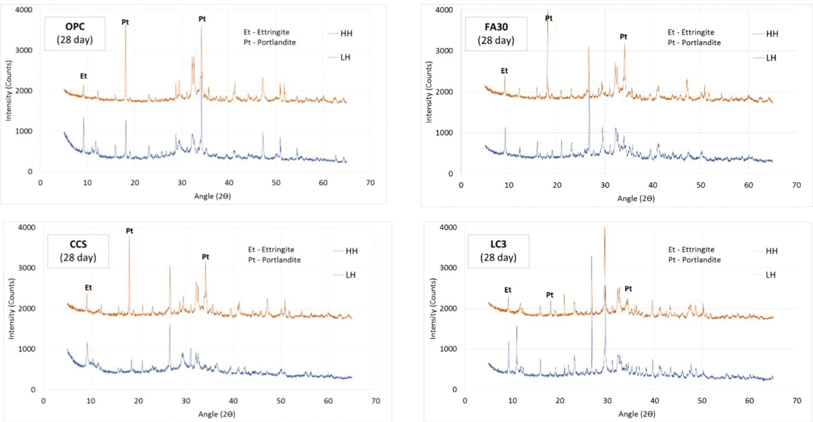

Fig. 2. XRD pattern of hydrated cement pastes at 28 days for both LH and HH

4 Conclusion

- It is evident the loss of strength occurs due to improper curing in all blends irrespective of clinker replacement factor.
- Both RH and curing period affects the strength development. This is more predominant in OPC where the clinker factor is high. The hydration of clinker phases especially belite, is highly influenced by presence of water in the system.

- It is to be noted that blended cements including FA30 has shown almost similar trend. This can be due to the particle size distribution fly ash and slag. The fine nature of these materials increases the pore refinement. The refinement of pores at specimen surface at early ages helps to maintain internal RH, by stopping evaporation during later age hydration.

The internal RH of specimen can impact the effect of curing, leads to possible influence of specimen size. Further work is required to check the influence of w/b ratio. Once the surface pores of the specimen stop percolation, the influence of external RH on properties of specimen as a whole stops. The time of occurrence of this transformation, after which curing has no influence, is depends on physical and chemical properties of raw materials, temperature history, w/b ratio etc.

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