COMPARATIVE EFFECTIVENESS OF VARIOUS CONVENTIONAL AND MODERN CURING TECHNIQUES IN CONCRETE

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DEDICATED

TO

MY PARENTS AND SIBLINGS

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In the name of Allah, the most merciful, the most compassionate all praises be to Allah, the lord of the worlds and prayers and Peace be upon Muhammad his servant and messenger.

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ABSTRACT

Appropriate curing of concrete is of vital importance in development of desired material properties in concrete namely compressive strength and durability based on uniform dense microstructure. Improper and intermittent curing is considered as one of the major reasons for concrete failures as evident in the form of cracks that consequently lead to low compressive strength and durability issues in concrete structures. Conventional concrete practices require cost efficient and effective curing methods be followed to achieve strength and durability with available materials at site.

An experimental program was designed to study the behavior of concrete under various conventional and modern curing techniques. One hundred and eighty concrete cylinder specimens were investigated with different curing techniques namely, water curing, burlap curing, liquid membrane-forming compound curing, combinations of admixture and liquid membrane-forming compound curing, admixture and burlap curing compared to air curing. In addition, new internal curing methods namely light weight aggregate, super absorbent polymers, and shrinkage reducing admixtures were investigated to quantify their effects on curing of concrete. Comparison of microstructure development with scanning electron microscopy (SEM); quantitative analysis with energy dispersive spectroscopy (EDS) analyses and compressive strength at different ages were conducted to monitor the effect of various curing methods on microstructure and strength in concrete. The results indicate that development of microstructure differs based on variation in chemical composition that occurs during hydration processes from various curing regimes.

The results exhibit higher attainment of compressive strength in case of wet burlap curing and immersed curing than rest of the compared techniques. Internal curing methods of light weight aggregates and super absorbent polymers were found to be excellent in reducing autogenous shrinkage in concrete due to uniform distribution of curing water within the mix; consequently leading to uniform microstructure and superior concrete for construction purpose. This work is helpful in establishing the best curing techniques to obtain higher compressive strength and durability in concrete. The results are also useful in determining appropriate curing technique to suit specific field conditions.

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CHAPTER 1

INTRODUCTION

1.1 General

Curing is a process where concrete is protected to keep the moisture and maintained within a reasonable range of temperature to allow cement hydration and pozzolanic reactions so that its potential properties of mixture can be developed (ACI 318-14, 2014). This process results in concrete with increased strength and decreased permeability (high durability). If during the curing process, ambient temperatures of concrete are within acceptable ranges, only the moisture content requires to be controlled. Curing of concrete has always been a challenge and wide range studies have been performed to evaluate effectiveness of curing techniques. In areas where shortage of water, lower humidity and higher winds are potential problems, sustainability of water may be achieved using appropriate liquid membrane forming compounds and various chemical compounds for curing of concrete as stipulated in ACI 305 (2010). Curing also plays significant role in mitigating micro cracks that severely affect durability. Effective curing becomes problematic in hot weather since high ambient temperatures and low humidity lead to evaporation of mix water. Drying winds also increases the rate of evaporation further contributing to plastic shrinkage cracking. It becomes even more challenging when concrete is mixed with water demanding supplementary cementitious materials such as Silica Fume, Fly Ash, and Ground Granulated Blast Furnace Slag and is subjected to non-conducive hot environments immediately after placing and consolidation.

Portland cement concrete is a composite material where aggregates are added and mixed in a porous system of hardened cement paste. The reaction between cement and water includes physical and chemical processes known as hydration of cement. If the amount of water added initially into the concrete sustains sufficient amount of hydration to develop desired properties of a given concrete, curing measures becomes necessity to ensure this water remains in concrete till desired properties are attained. The concrete surface should be protected against loss of moisture at once following finishing process. As the hydration process continues, the durability and strength of the bonded particles increase and porosity between these particles decreases. If observed through scanning electron microscope; a fine

detail of inter particle bonding and development of hydration products and partially hydrated products are shown. Since hydration requires the availability of water and proper curing environment. If there is not enough internal bleeding water cement particles tend to remain unhydrated and crystalline microstructure will not form a strong bond we need as shown in the Fig. 1.1that calcium hydroxide crystals are not formed in this matrix.

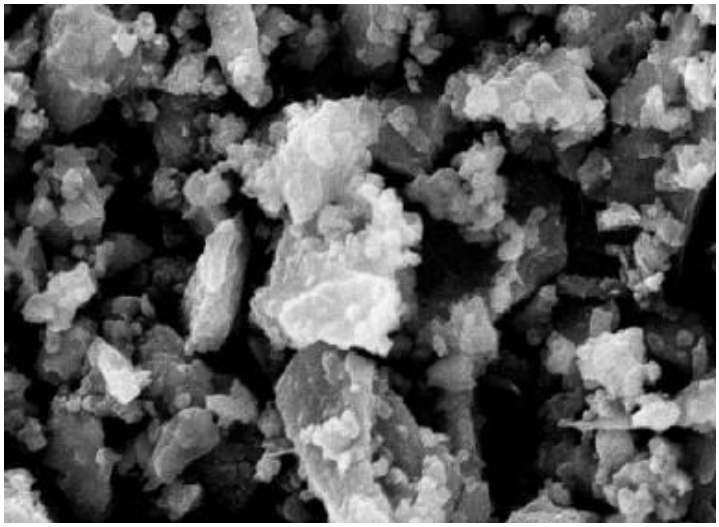


Fig. 1.1 At magnification of 2000x- Some unhydrated particles (ACI-308)

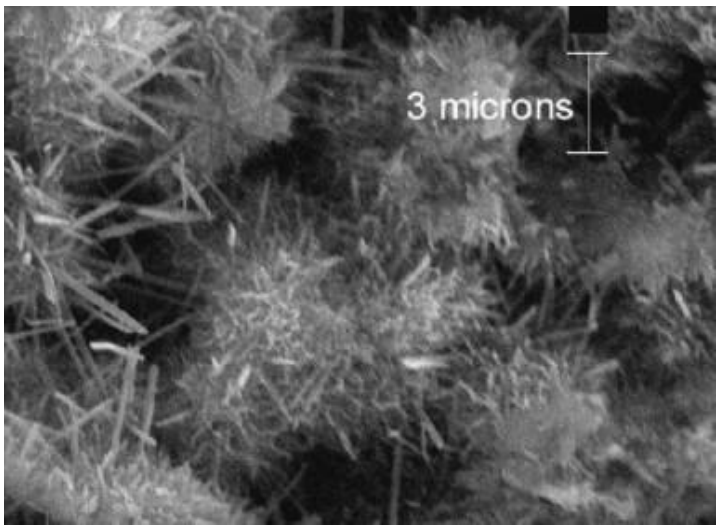


Fig. 1.2 At magnification of 4000x- Partially hydrated Portland Cement Particles (ACI-308)

In Fig 1.2 multiple hydrated products are shown. This shows that structure is not homogenous; some of the areas are found to be dense while other areas seem porous.

Substantial surface drying can happen when curing requirements are delayed until the entire structural element is finished because highest rate of evaporation from concrete surface occurs following last pass from the finishing tool. Concrete is allowed to dry out rapidly leading to significant early age drying shrinkage cracks. Insufficient curing happens to be one of main factors contributing towards powdery and weak surfaces, and low resistance. These are related to the interconnection pores and capillaries in cement paste. While the volume of the capillaries and pores in cement paste are related linked with w/c ratio, they are also indirectly linked with the extent of water curing. With the passage of time, water curing leads to hydration products to fill the pores and capillaries present and thereby reduce the porosity of the cement paste.

Curing takes place after placing and finishing of concrete is done. It involves keeping the desired moisture and temperature conditions maintained for extended period. Sufficiently cured concrete specimens have adequate moisture for hydration and development of compressive strength, durability, volume stability, abrasion and scaling resistance and resistance to freezing and thawing.

The length of optimum curing time depends upon mixture proportioning, specified compressive strength, volume of concrete elements and ambient weather conditions. Pavements, parking lots, sidewalks, floors, driveways, canal linings and structural concrete members such as bridge decks, columns, piers, slabs, beams, footings, cast-in-situ walls, retaining walls demand a minimum curing period of 7 days for ambient temperatures over 40 F. American Concrete Institute (ACI) Committee 301 stipulates a minimum curing period corresponds to concrete achieving 70 percent of specified compressive strength. The often specified 7-day curing commonly corresponds to approximately 70 percent of the specified compressive strengths. The 70 percent of specified compressive strength can be reached when concrete is cured at higher temperatures or when specific admixture combinations are employed. Similarly, for lower curing temperature, longer time may be required for various material combinations. For this reason, ACI Committee 308 stipulates following minimum curing periods:

- ASTM C 150 Type I cement 7 days
- ASTM C 150 Type II cement 10 days
- ASTM C 150 Type III cement 3 days
- ASTM C 150 Type IV or V cement 14 days

1.2 Objectives

This research aims at identifying the efficient and effective technique out of all curing methods in practice, that requires lesser time, has the tendency to develop better microstructure for durability and strength of concrete for regions where there are high ambient temperatures, high drying winds and low relative humidity. Permeability is a significant parameter which is directly associated with microstructure and durability of concrete. The effect of development of microstructure on concrete durability performance is mandatory to establish the most efficient curing regime. Scanning electron microscope (SEM) has been used for microstructural analysis of concrete and its component. While describing the microstructure of concrete, overall distribution of solid phases such as hydrated cement particles and porosity is taken into account. Since each hydrated product plays its own part in strength development, SEM proves to be an insightful tool to aid in correlating the development of these products with respective compressive strength results. With this in mind, various curing techniques were employed to evaluate the development mechanism hydrated products and address them through compressive strength results. According to ACI 318-14 (2014), compressive strength for 28-days specimens, cured and protected to reflect good field practices, should not test less than 85 percent of standard laboratory moist cured specimens. This rational basis needs to be validated using various conventional and emerging curing techniques on normal strength concrete. Since normal strength concrete is an integral part of research domain in construction industry as well as being implemented widely in the construction projects all over the world, therefore through this research program, effects of curing regimes shall enable a better understanding of an efficient methodology for improved durability and strength. Moreover, to identify and establish quality of concrete microstructure that helps improve durability in concrete using different curing techniques, detailed study on microstructure development in concrete is also desired. The main objectives are stated below:

- According to ACI – 308, compressive strength for 28-days cylinders, cured and protected to reflect good field practice, should not test less than 85 percent of standard laboratory moist-cured cylinders. This rational basis must need to be validated using various curing techniques.
- To evaluate the most efficient curing regimes and associated durability performance can be most beneficially determined from the development of microstructure through various degree of hydration.
- Scanning electron microscope (SEM) and electron dispersive spectroscopy (EDS) imaging and analyses shall be conducted on concrete samples cured using various curing techniques to establish the efficiency of different curing techniques.
- Comparing the compressive strength results of various curing techniques on specified days to find out the best possible curing techniques among them to be identified for construction practices.

To accomplish the above mentioned objectives several tasks were undertaken:

- Conduct literature review.
- Develop the test setup.
- Casting the cylindrical specimen.
- Conducting the compressive strength testing on specified days.
- Conducting SEM analysis.
- Process the EDS test data to develop relationship between strength and days.
- Evaluate the experimental results.
- Develop conclusions and recommendations.

1.3 Organization of the report

- Chapter 1 is an introductory chapter about importance of curing, objective of the study and thesis overview.
- Chapter 2 represents brief literatures review on various curing techniques and results is discussed.
- Chapter 3 represents the procedure and materials of test setup, the testing facility and construction of specimens. The testing apparatus and data acquisition system is also elaborate.

- Chapter 4 discusses the tests carried out, the observations, test results and evaluation of test results.
- Chapter 5 gives the conclusions based on findings of this study and recommendations for further studies are presented.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Curing of concrete is one of most significant practices that promise a long term benefit in terms of durability of a structure. Due to intense and very significant advantages, curing has been the pivotal point of several research works during past few years. This chapter contains notable studies carried out on various curing techniques implemented on concrete.

Previous studies have taken into account many different variables in this aspect. Some of these variables are type of admixtures, liquid membrane-forming compound compounds, mode of curing and days of curing to quantify the effect of curing on concrete.

2.2 Previous studies on curing practices

Much research has been carried out on finding the optimum technique for curing of concrete. Various materials and compounds have been used to analyze their behavior in the strength development of concrete namely acrylic-based liquid membrane-forming compounds, water-based curing compounds, and superplasticizer. With an aim to achieve desired compressive strength and rapid construction in the field practices, liquid membrane-forming compound compounds have been used extensively to study their effects. These compounds are highly effective in reducing drying and plastic shrinkage cracks as studied by Al-Gahtani (2010). He prepared specimens with Type-I, fly ash and silica fume added cement concretes. These specimens were cured using wet burlap and through application of liquid membrane-forming compounds of two types namely water and acrylic based curing compounds. The objective of this study was to keep the concrete specimens wet enough to assist the hydration process. Specimens were mixed and prepared by plain cement, silica fume added, very fine fly ash (VFFA) added, and fly ash added cement. The study indicated that liquid membrane-forming compounds can be employed in conditions where water curing is not available. Among the investigated compounds namely acrylic based and water based curing compounds, acrylic based compound performed better than the other compound. In Fig. 2.1 compressive strengths after 90 days of plain and blended cements are shown. Both water based and acrylic based curing compounds were found effective in reducing plastic and drying shrinkage strains in plain and

blended cements. Moreover, efficiency of curing with regards to specified compressive strength was in the range of 84-96%. Combination of curing compounds with chemical admixture in the concrete mix has also been investigated in terms of their effects on the development of concrete and to facilitate the curing conditions of concrete as demonstrated by Al-Gahtani (2010).

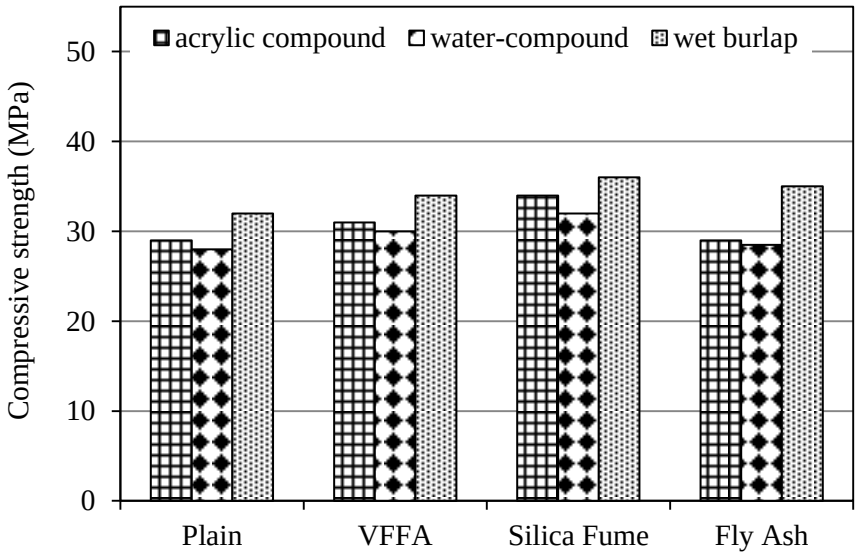


Fig. 2.1 Compressive strength of cement based mortar under various self-healing conditions (Al-Gahtani (2010))

Chemical admixtures such as cold weather admixtures, set retarding admixtures and water proofing admixture together with high performance superplasticizer have been inspected with acrylic, hydrocarbon or dispersion based liquid membrane-forming compounds.

Compressive strengths of concrete specimens at 28 days produced with OPC with 10% silica fume replacement, Portland pozzolana cement (PPC), and Portland cement (OPC) cured using conventional curing is observed to be more than the specimens cured using curing compound as studied by (Krishna et al., 2010). The experiment involved testing of 99 test specimens of size 150 x 150 x 150mm covering the three parameters namely the type of curing, type of cement, and age of curing. Authors observed reduction in compressive strength at all ages for specimens with PPC and OPC with 10% silica fume in it when compared with OPC specimen. Specimen cured using curing compound produced similar results as produced by conventional curing with OPC whereas reduction in percentage loss was recorded in specimens of OPC with 10% silica fume and PPC. The results achieved were encouraging for concrete mix prepared with OPC and OPC prepared with 10% SF. 3rd day compressive strength of concrete with 10% replacement of

silica fume and cured with liquid membrane forming compound is 8.54% more than the OPC. The 28th day compressive strength of concrete was reduced down by a percentage of 9.76 and 3.31 for 10% replaced silica fume mix and by a percentage of 11.75 and 11.41 for PPC in comparison with OPC concrete mix when it was conventionally water cured and cured using liquid membrane forming curing compound. At all ages, PPC concrete mix was found to have lower compressive strength than OPC and 10% replaced with SF concrete mix. Fig. 2.2 shows the behavior of concrete specimens at various ages of curing.

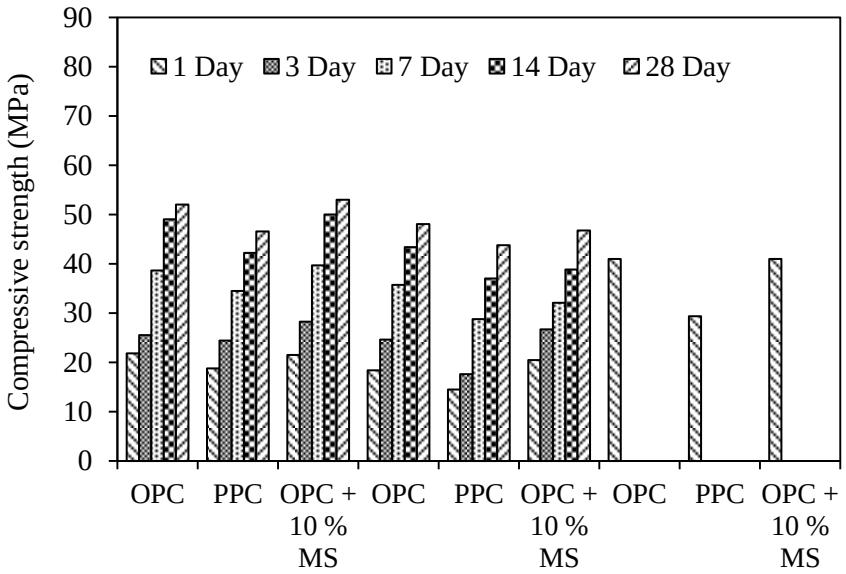


Fig. 2.2 Comparison of compressive strength of various curing methods (Krishna et al., 2010)

For high strength self-compacting concrete conventional curing technique such as water immersion method produces the most optimum results. Qureshi et al. (2010) examined the effects of curing compound on high strength self-compacting concrete and applied conventional techniques such as water immersion in laboratory, sprinkling water in a field condition and using curing compounds. The authors employed three difference curing techniques to monitor and evaluate the most optimum curing method. Controlled specimens were stored for 24 hours at a relative humidity 90% and temperature of 20 Celsius. After this, the cubes were cured in a temperature controlled water bath up to their testing. A total of 24 specimens were cast and exposed to on field conditions cured by water sprinkling along with curing of some on ground full scale concrete members. Same number of specimens were cured by a curing compound at a rate of 5 m^2/liter on the surface. A substantial difference of strengths was obtained as the day

progressed indicating the highest strength development in case of controlled specimens as shown in Fig. 2.3. Specimens cured using curing compounds exhibited better strength development than on-field curing compounds. The authors concluded that water immersed specimen provided the best compressive strength results in case of high strength self-compacting concrete.

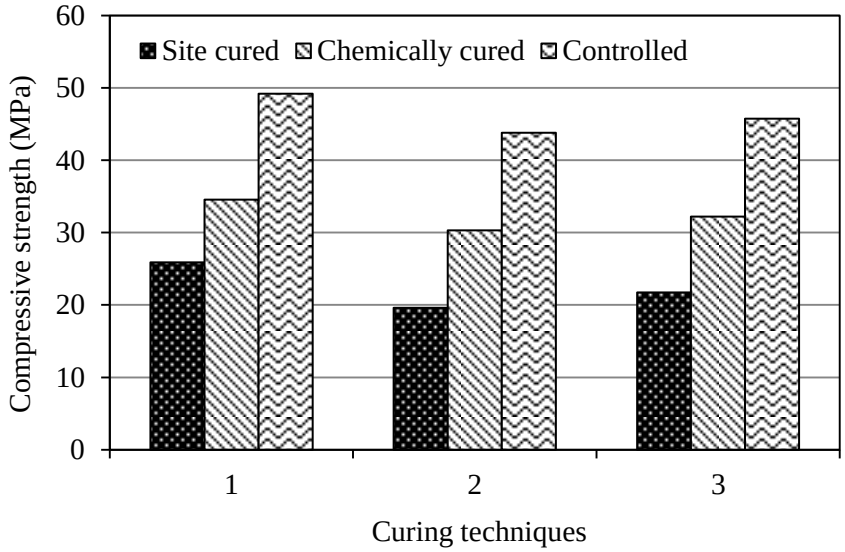


Fig. 2.3 Comparison of compressive strength of various curing methods at 3, 7 and 28 days (Qureshi et al. (2010))

Similarly, Raheem et al. (2013) applied conventional curing practices and concluded that specimens cured using moist sand produced the highest compressive strength results among the various techniques namely spray curing, water-submerged curing, air curing, moist sand curing, polythene curing, and burlap curing in normal concrete. He also concluded that all methods except for air curing easily produced code specified minimum compressive strength.

Moisture content in the concrete plays effective role in the hydration process as well as the ultimate strength development. Popovics (1986) explained how distribution of moisture content affects compressive strength of the concrete rather than overall moisture content. He applied air curing, moist curing and air plus moist curing and accelerator produced concrete specimens. He concluded that the highest compressive strength of concrete specimen produced at any age was using moist curing followed by a finishing 3-day air curing prior to compressive strength test. Moisture gradient in the concrete appreciably effects the concrete strength in accordance with (ASTM C42 / C42M, 1990) and (ACI Committee 318, 1989). Bartlett and MacGregor (1994)

examined the differences between compressive strengths of soaked and air dried concrete cores for better microstructure of the structural element and consequently better compressive strength. They added that interior surface does not undergo any change thereby it restricts the swelling of outer surface causing a residual stresses which in turn decreases the compressive strength. However drying the specimen causes shrinkage at the surface and increases the compressive strength. They concluded that strength of air dried cores comes out to be on average 14 percent larger than strength of soaked cores.

Influence of cylinder size and curing conditions on normal and high strength concretes is significant. Air cured cylinders has compressive strength aproximately 16 percent lower than sealed cylinders Aitcin et al. (1994). They concluded that the water cured cylinders resulting in better microstructure development gave higher compressive strengths than the sealed cylinders which in turn gave higher compressive strength than the air cured cylinders.

Initial water curing on concrete specimens has significant effect on compressive strength. Ozer and Ozkul (2004) determined the effect of initial water curing on the compressive strength of concrete by applying six different curing regimes mainly included continuous water storing, air storing and four different regimes under varying curing periods of 3,7,14 and 28 days respectively. They concluded that concrete formed with pozzolanic cement showed consistent strength gain over the testing ages due to pozzolanic properties that improved microstructure development in contrast to concrete formed with OPC. Another curing technique that satisfies the strength criteria in lesser time is accelerated curing. The authors studied the influence of curing with water on strength development of ordinary Portland cement concretes and pozzolanic cement concretes. Six different curing regimes were employed at three different type of cement concretes namely two pozzolanic cement (blended and trass cement) and ordinary Portland cement. The development of compressive strength indicated that curing environments effected the compressive strength of both pozzolanic cement and OPC concrete however, improper curing practices effected more in case pozzolanic cement than OPC. For pozzolanic cement concrete specimens, 7-day water curing was mandatory to initiate the pozzolanic activities. It was found that blended cement concrete and trass concrete specimens achieved the strength range of OPC concrete specimens continuously water cured, in a short period of 2 months when former were water cured for 14 days. However, when blended and trass concrete specimens were moist cured

for 3 days or lesser period, these specimens failed to achieve the strength of OPC concrete specimens. Moreover the strength gain rate of OPC concrete specimens under various curing methods is lower than blended and trass concrete specimens although this holds opposite when all of them was air-cured. Lastly, OPC concrete specimens show reduction in strength between 90 and 180 days expect for 14-days or water immersed specimens. Alternatively, pozzolanic cement specimens showed increase in strength after 90 days which can be attributed to the pozzolanic activities over the shrinkage cracking. Efficiency of concretes mixed and prepared by low early strength cement increases when curing temperature is increased as experimented by (Ozkul, 2001). The authors investigated the effects of accelerated curing on concrete samples prepared from OPC and blended cement using warm water and boiling water. The result showed that compressive strength development is greater for concretes produced with OPC than that of blended cement (trass cement) as shown in Fig 2.4, Fig 2.5 and Fig. 2.6.

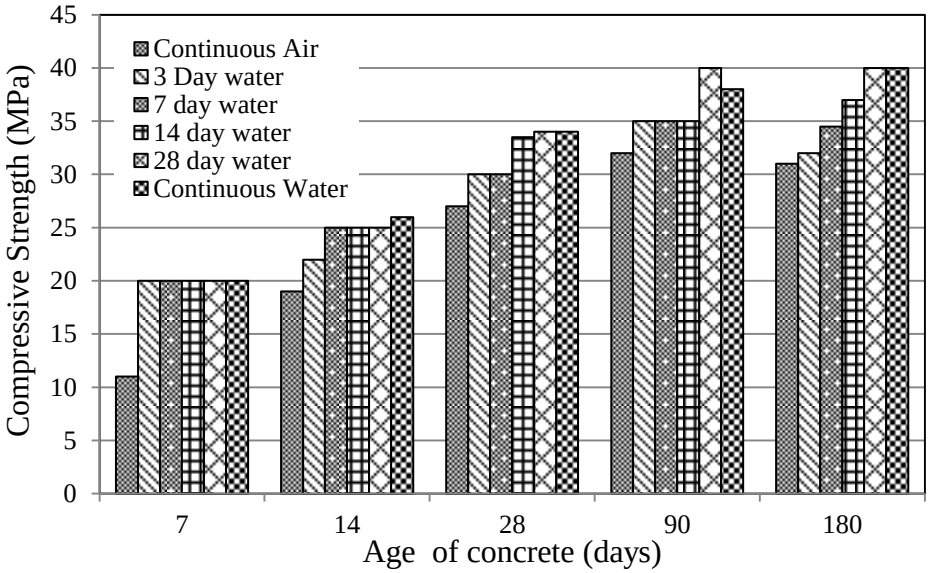


Fig. 2.4 Strength development for OPC concretes (Ozkul, 2001)

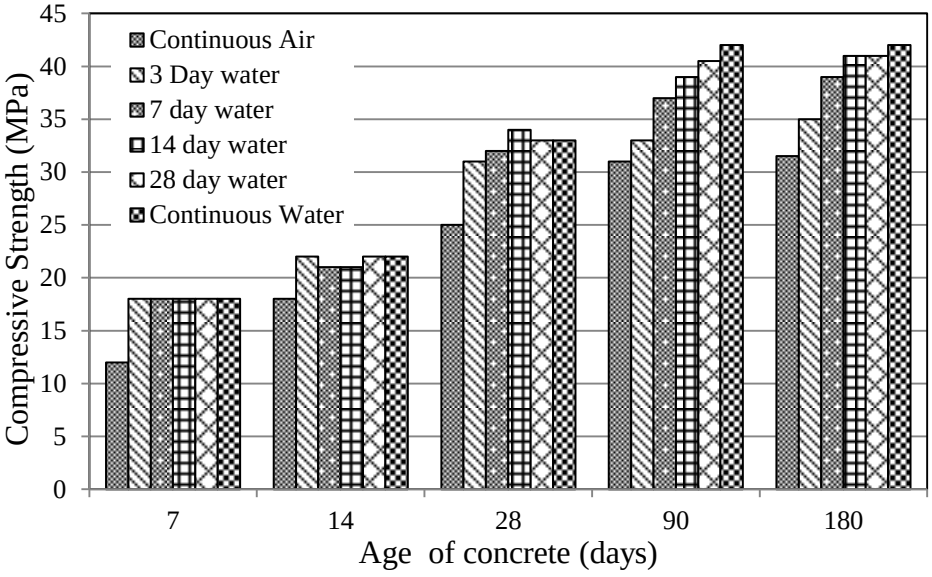


Fig. 2.5 Strength development for BC concretes (Ozkul, 2001)

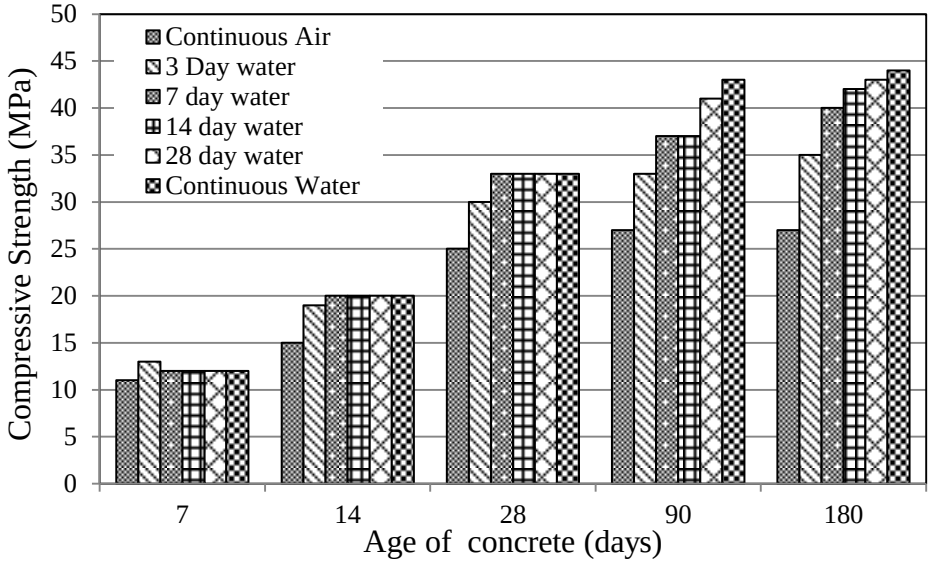


Fig. 2.6 Strength development for TC concretes (Ozkul, 2001)

Apart from the abovementioned curing regimes and techniques recently discussed, internal curing is a promising technique trending these day in concrete construction. Concrete mix is tailored by adding suitable curing agents that can augment the curing requirements of concrete. High cement contents and low w/c ratio makes concrete vulnerable to self-desiccation and autogenous shrinkage which can be benefitted from added moisture provided by internal curing indicated by Cusson et al. (2007). Presently, implication of this technique mostly involves adding

light weight aggregate (LWA) and super absorbent polymers (SAP) as curing agents. These materials have proven significantly important in absorbing a large quantity of water and serving as water reservoir inside the mix to replenish the water requirement for continuous hydration and thereby reducing autogenous shrinkage resulting from self-desiccation.

Internal curing is state-of-the-art technique that helps concrete attain maximum compressive strength without external curing. Fine light weight aggregate as internal reservoir with internal porosity improves hydration of cementitious materials more than water curing at specimen surface Siddiqui et al. (2013). Higher dosages of these light weight aggregate increases the rate of hydration. Moreover, they concluded that curing compounds provide increase hydration of cement paste in a sealed environment, attributing to high water content, same as that of water-cured specimen. Distilled, lime-saturated and cement pore water solutions were used to investigate the effects of curing. The effects of dry cured using fine light weight aggregate materials as internal agent proved highly effective in attaining 28 days compressive strength as compared to chemically cured specimens as shown in Fig. 2.7.

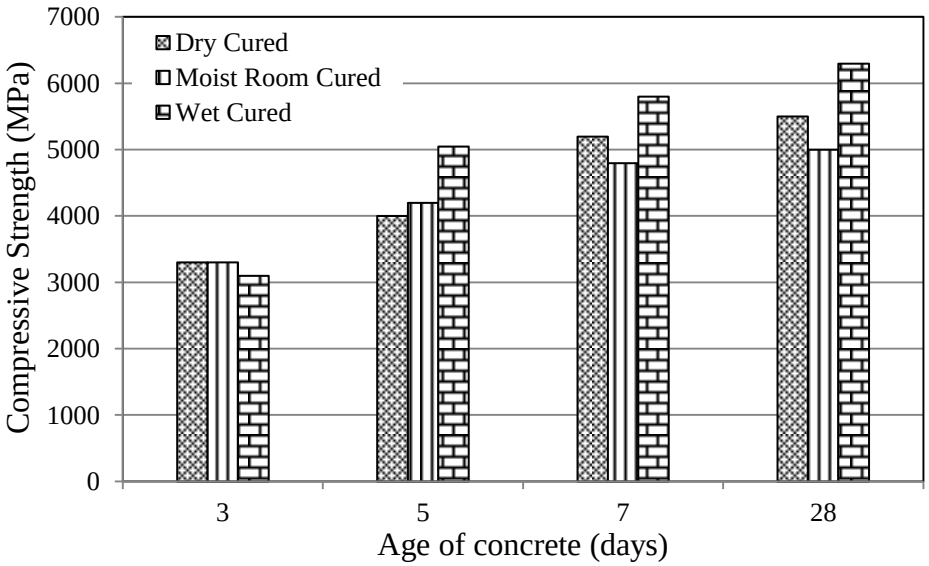


Fig. 2.7 Concrete cylinder strength development under different curing conditions (Siddiqui et al. (2013))

One of the notable works on internal curing was carried out by Bentz and Stutzman (2008) where light weight aggregate was used as curing medium to support the internal curing. Three different blended cements were used having 20% slag, 8% silica fume, and 25% fly ash by mass

respectively. The compressive strength results measured showed reduced strength in all the cases of internally cured mortar cubes on third day testing when compared with their counterparts. However, with the passage of time, the condition improved and on 56th day the compressive strength of all internally cured specimens resulted in better performance than the conventional techniques, offsetting any reduction in strength due to use of curing agent as shown in Fig. 2.8.

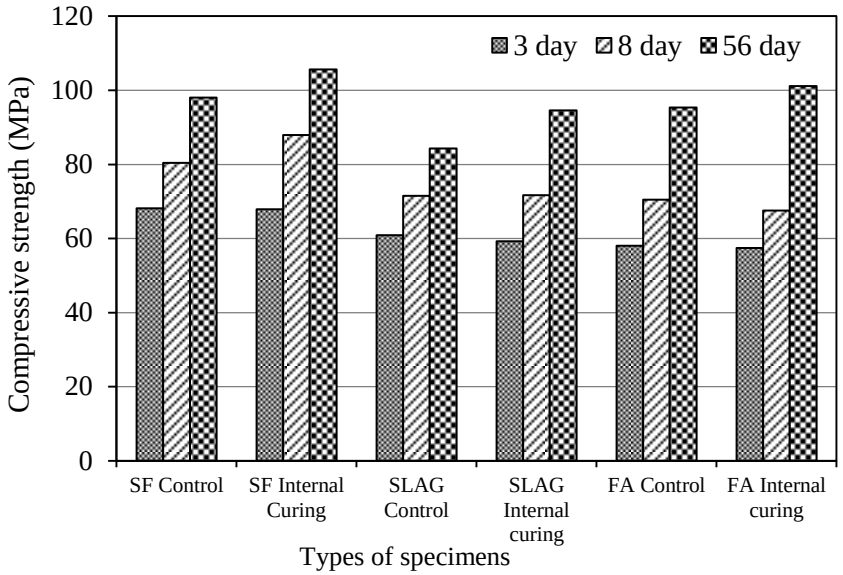


Fig. 2.8 Compressive strength development for blended cement concrete (Bentz and Stutzman (2008))

Bentz and Snyder (1999) started off their working on developing an equation that estimates the replacement range required to ascertain sufficient water demand for thorough curing. A 3D continuum microstructural model was prepared and through aided computer simulations it was suggested that a well dispersed matrix of light weight aggregate has a favorable impact on the curing of concrete. Dayalan and Buellah (2014) introduced expended shale as internal curing agent in 10%, 15%, 20% and 25% proportions as replacement of coarse aggregate. They concluded that expanded shale increased degree of hydration developing a dense microstructure. Compressive strength results at 7^{th} day came out to be lesser than conventionally cured specimens however ensued strengths at 21^{st} and 28^{th} days revealed greater performance with regards to compressive strength against its counterparts as shown in the Fig. 2.9.

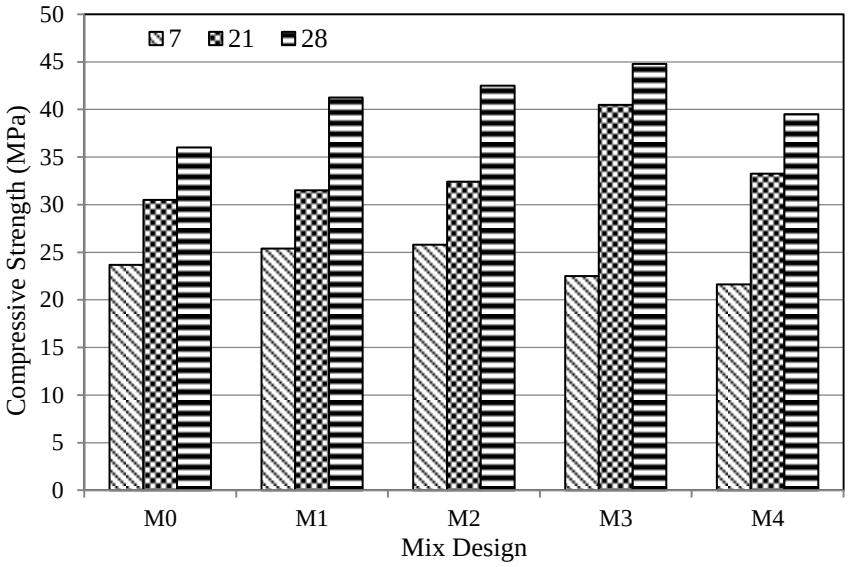


Fig. 2.9 Comparison of average compressive strength on 7, 21 and 28 days for various mixtures. (Dayalan and Buellah (2014))

Gifta et al. (2013) introduced super absorbent polymers (SAP) and lightweight aggregates (LWA) to process the curing in the high performance concrete specimens. 0.3% of SAP and 25% of LWA was employed in the mix. Results in Fig. 2.10 indicated that LWA samples exhibited an increase of 12.35% of compressive strength while SAP exhibited about 6.88% increase than the control mix. This demonstrated that use of LWA has proven to be effective than SAP.

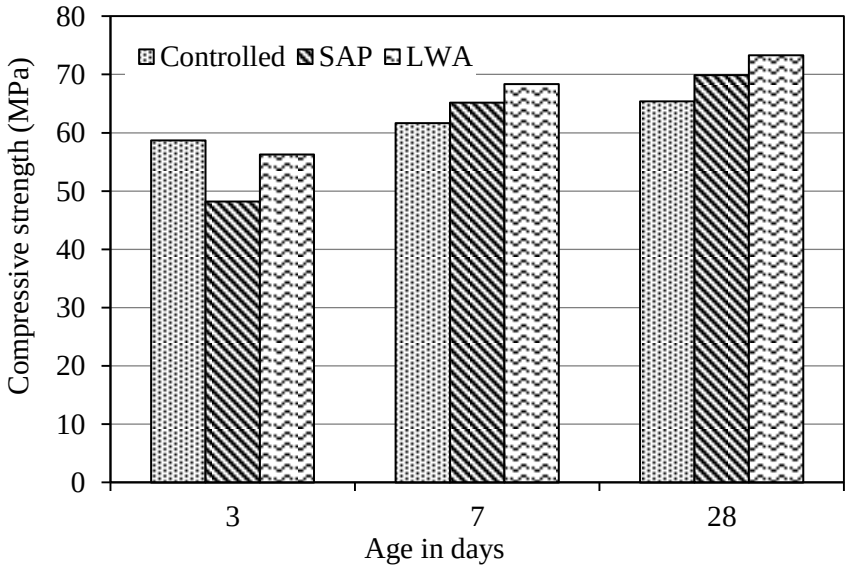


Fig. 2.10 Comparison of compressive strength on 3, 7 and 28 days for different mix (Gifta et al. (2013))

Olawuyi and Boshoff (2013) also studied the effects of addition of SAP on the compressive strength of concrete. They concluded that with higher contents of SAP in concrete mix lower will be the density and consequently higher the capacity. Based on their studies, they evaluated that quantity of SAP 0.3% by weight of cement is limited to obtain the most effective results. SAP was introduced in two different proportions in high performance concrete specimens of 100mm cube and tested after being cured in water for 3, 7, 21 and 28 days. The results indicated a reduction in compressive strength as the SAP contents in the mix are increased at a rate of 0.4% however, a strong deviation from the trend was observed at 0.6%. The 28 day strength of high performance concrete specimens containing both the SAP contents was observed to fulfill the minimum specified strength criteria as shown in Fig. 2.11.

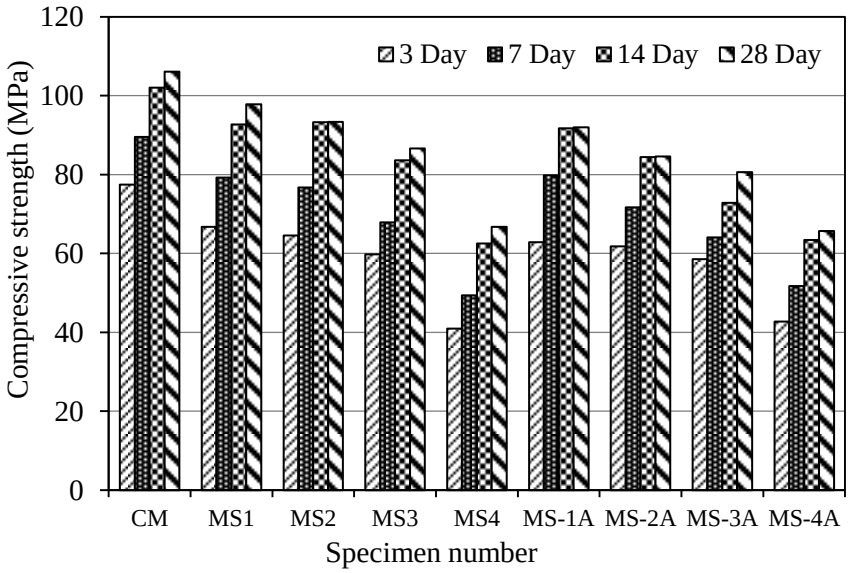


Fig. 2.11 Compressive strength of HPC with SAP (Olawuyi and Boshoff (2013))

Although the internal curing is still incipient and its evaluation under progress, foregoing studies have clearly indicated the significant importance of the employed curing techniques and their effects on the durability and compressive strength of concrete. LWA has substantial effects in terms of achieving high strength concrete with minimum strength and quality loss while SAP is considerably a new concept and requires more deliberate efforts to demonstrate a clear correlation between internal curing and autogenous shrinkage affecting the compressive strength.

For quality and durability purpose in concrete, it is imperative to understand the significance of curing as illustrated by several studies. Various techniques have been investigated namely water

immersion, curing compound, sealed curing, air curing, accelerated curing and internal curing. All these curing regimes have addressed the sensitivity of concrete to hydration and provided favorable results; however, there is large variation in results exhibited by these techniques. Liu et al. (2014) explained that connectivity of pores is strongly influenced by w/c ratio, curing conditions and alkali content of cement. Curing for strength and curing for durability may require different practices to promote the development of microstructural features as may be seen from the studies conducted by Bentz and Stutzman (2006). More state-of-the-art methods are under consideration and being investigated so as to quantify the most efficient and effective curing technique.

CHAPTER 3

EXPERIMENTAL PROGRAM

3.1 General

From the stability and safety point of view, structures are prone to the earthquake worldwide. Researchers and engineers all around the world have analyzed the damage of buildings which experienced the Earthquake and it has been found out that elements that were damaged and resulted in collapse of the buildings had compressive strength lesser than the specified/targeted strength which describes lack in curing practices of structural elements. Improper curing is considered as one of the most significant and notable reasons for concrete failures in structural elements evident in the form of cracks easily notable by naked eyes. To quantify the best curing technique on different specimen compressive strength tests were conducted along with scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) analyses and results were compared. Description of specimen for these tests is provided in the Table 3.1

Table 3.1 – Curing techniques and sample description

Sr. No	**Specimens**		**No of cylinder specimens**				**Sample dimensions for compressive strength test**	
							Dia (in)	**Height (in)**
1	Air cured (NC)	C1	3 days	7 days	14 days	28 days	6	12
2	Water immersed (WC)	C2	5	5	5	5	6	12
3	Liquid membrane-forming compound cured (CC)	C3	5	5	5	5	6	12
4	Burlap water cured (BC)	C4	5	5	5	5	6	12
5	Combination of admixture and liquid membrane-forming compound cured (ACC)	C5	5	5	5	5	6	12
6	Combination of admixture and burlap curing (ABC)	C6	5	5	5	5	6	12
7	Light weight aggregate (LWA)	C7	5	5	5	5	6	12
8	Super Absorbent Polymers (SAP)	C8	5	5	5	5	6	12
9	Shrinkage Reducing Admixture (SRA)	C9	5	5	5	5	6	12

Curing is the key to prevent much of the mixed water from evaporating before required hydration can be achieved (ACI-308, 2001). To establish comparative effectiveness of various curing techniques in concrete, an experimental program was designed to study the progress of hydration products in concrete and to establish compressive strength development in concrete at four target ages with nine curing techniques. To quantify various hydration products through microstructural analyses of concrete, a detailed SEM/EDS analysis technique was used. Specimens of 6x12 inches standard cylinder size 20 in numbers of each regime were casted. Five specimens for each regime on specific testing day were tested to effectively assess the compressive strength results. Specimens name indicate the techniques and methods used to quantify the compressive strength results and SEM analysis. Controlled specimens containing no admixture and chemical compound are denoted by "WC". These specimens were placed in the water pond after demoulding and removed for testing on specified age. Whereas, "NC" indicates that specimens were cast and left open under covers without any curing applied on them, also referred to as air curing. "CC" specimen shows the chemically compound curing which was applied at a rate specified by the manufacturer i.e. $5m^2$/liter. "BC" specimen represents the burlap curing where hessian cloths were wrapped around the specimen in two fold and were cured three times a day. "ACC" specimen signifies the incorporation of superplasticizer in the mix at 1% of weight of cement and then cured through chemical compound. In the same way, "ABC" stands for the specimen with superplasticizer incorporated and curing was performed with burlap curing. For internal curing three different curing techniques were employed. "LWA" indicates addition of pre-wetted lightweight aggregate as internal curing agent with absorption capacity of 11.6 % having w/c ratio 0.36. "SAP" is another internal curing agent used for internal curing of specimens. SAP works in cross-links (chemical structure of polymers) that prevents polymers strands from dissolving and provide pores for water. Sodium polyacrylate having absorption capacity of 300 times of its own weight was used for such purpose at 2g per kg of cement proportion. Additional mixing water of 13.5 liters was added to the mixture in an amount sufficient to saturate SAP particles. This amount corresponds to entrained w/c ratio of 0.03 for 0.2% of weight of SAP in the concrete mix. Lastly, "SRA" is a shrinkage reducing chemical used for internal curing at 1% of weight of cement. Sample drying for SEM examination is needed. For this purpose, it was oven dried at 40^oC. The sample is placed on 12 mm aluminum stub rested on the plate. For this reason, sectioning of the sample is done so that it rests perfectly

on the stub. Small nylon brushes were used to sweep away dust and particles on the surface of specimens. Aluminum and copper conductive tape, double-sided and coated with organic adhesive is stuck to stub so that sample may be attached. Surface coating with gold, being conductor, was done on the sample. Once all the pre-requisites were done, the stub is mounted in SEM using mechanical attachment for SEM and EDS analyses.

The tests for compressive strength were conducted according to criteria defined in ASTM C-39 (revised 2014) and ASTM 2809 standard was followed during the process of scanning electron microscopy. Quantitative analysis by energy-dispersive spectroscopy (EDS) was carried out in the light of procedure defined in ASTM E1508. This chapter describes the experimental program including testing procedures. Details about material properties, sample preparation and testing methodology have also been included in this chapter.

3.2 Materials

The constituent materials used in this study were tested conferring to their relevant standards and to ensure consistency in their supply, periodical quality assurance tests were carried out. Given below are the tests conducted on different materials according to the guidelines provided by standards.

3.2.1 Liquid membrane-forming compound

One of the curing techniques that is widely being adopted these days is chemical curing. In this study, liquid membrane-forming compound was introduced at two different ways to cure specimen. For this purpose, Masterkure 107i water based concrete liquid membrane-forming compound has been used. This compound is membrane forming wax emulsion suitable to apply at freshly poured concrete. The resultant film retains sufficient moisture in the concrete to ensure full hydration of the cement essential for optimum strength development. Membrane cured concrete is typically harder and exhibits a dust free surface with a reduced incidence of drying shrinkage cracks. The recommended rate of application is 5-6 square meters per liter.

3.2.2 Light weight aggregate

Using lightweight aggregates (LWA) was studied as an internal curing agent used in concrete mix to evaluate the optimum method of curing. Fig. 3.1 shows the LWA as a crushed brick form (Khaaka) used in this study for the incorporation of water repository traits in the concrete mix.

Fig. 3.1 Light weight aggregates

Water absorption test was also conducted on LWA to determine the exact amount of LWA to be used. To evaluate the water absorption in lightweight aggregate 500 gm of LWA were submerged in water to make it saturated. After 24 h, they were surface dried and their saturated weight was measured. These aggregates were later placed in oven and were dried for 24 h and dry weight was measured. The source of LWA was crushed masonry bricks, with aggregate size passing 4.75 mm and retained on 2.00 mm sieves. On basis of this test, water absorption of LWA was found to be 11.6%. According to aggregate absorption capacity of 11.6% at complete saturation, it required 230 kg/m^3 of dry LWA for w/c ratio of 0.36.

3.2.3 Sikament 520 BA (Superplasticizer)

Sikament® - 520 BA is a highly effective superplasticizer with a set-retarding effect for producing free-flowing concrete in hot climates. In addition, it is a substantial water reducing agent for promoting high early and ultimate strengths complying with ASTM C-494 Type G. Sikament ® - 520 BA is used wherever high quality concrete is demanded under difficult placing and climatic conditions. Table 3.2 presents the technical properties of superplasticizer provided by sika. Superplasticizer was used in the amount of 1% of total weight of cement contents to ensure appropriate workability of concrete.

Table 3.2- Technical properties of superplasticizer

Chemical Base	Polymer type dispersion
Density (at 25°C)	Approximately 1.18 kg/lit
pH	8.0 – 9.5
Dosage	0.8 - 2.5% by weight of cement

3.2.4 Shrinkage reducing admixture (SRA)

These admixtures influence the residual stress development by effective reduction in surface tension. The purpose of adding shrinkage reducing admixture was to evaluate the comparative effects of SRA as an internal curing agent. This chemical is transparent in color and added in the concrete mix as 1% of weight the cement.

3.2.5 Scanning electron microscopy (SEM) analysis

A scanning microscope is provided for producing a scan image at high spatial resolution and in a low acceleration voltage area. SEM analysis shown in Fig. 3.2 is used for the examination of concrete and has proved to be an insightful tool for the microstructural analysis of concrete and its components. ASTM C1723 introduces the application of SEM/EDS analytical techniques for the examination and microstructural analysis of concrete whereas ASTM E1508-12a clearly defines that EDS, is used as quantitative analysis in conjunction with SEM. In this study, the volume fraction of constituents distinguished in SEM has been measured in quantitative analysis. The X-rays used for EDS analysis provides qualitative analysis to determine which elements are present, semi-quantitative analyses to identify the chemical compounds, quantitative analysis to determine what is the concentration of each element, and mapping the distribution & concentration of each element. The quantitative analysis depends on the mass concentration of element in the sample detected by X-rays. The percentages of major constituents in this study have been determined through EDS technique. The measured peak intensities are the reflection of concentration of elements, which can be translated into their characteristics in the strength development of concrete.

Fig 3.2 Scanning electron microscopy setup

3.3 Experimental procedure

The experimental setup and procedures for conducting scanning electron microscopy, Electron Dispersive Spectroscopy (EDS), and compressive strength tests on concrete elements are discussed here. Mix proportions for nine different concrete mixtures are presented.

3.3.1 Mix proportioning

While performing the experimentation, two sets of design mixes were prepared. One mix containing all the constituents including superplasticizer while the other mix was prepared without admixture. Batching was done by weight of materials using batching plant and slump was checked after batching out the concrete. The mix proportion was adopted as 1:1.63:2.45 and a W/C ratio of 0.45 for conventional mixes and 0.33 and 0.36 for SAP and LWA internal curing specimens respectively.

Table 3.3 – Mix design of different sets of specimens

Items description	Type	Actual by weight	Standard by weight	Unit
Cement	Bestway	31.5	450	Kg
Coarse Aggregate	Margalla Crush	77.35	1105	Kg
Fine Aggregate	Lawrencepur	51.52	736	Kg
Water	--	14.17	202.5	Lit
Admixture	Sikament 520 BA	0.32	4.62	Lit
LWA	Khaaka	19	230	Kg
SAP	Polyacrelate (Local Market)	63	900	Gram
SRA	Mapecure	0.315	4.62	Lit

The experimental program preparation of 120 cylindrical specimens using six different curing techniques namely water immersed curing, liquid membrane-forming compound cured, burlap curing, liquid membrane-forming compound curing with added admixture, burlap curing with added admixture and lastly a condition where no curing was done. The mixing procedure and time were kept constant for all the concrete mixes investigated and details of mixing proportions are presented in table 3.3.

3.3.2 Mixing and casting procedure

As per the given proportion, the quantities of cement, aggregate and water were determined by weight and trial batch was conducted using actual quantities as mentioned in the table 3.5. Once the trial batch was made and tested for 28 days to render satisfied results, mix was prepared for

research specimens. Cement, fine and coarse aggregates were added in the bins of batching plant. The concrete was mixed in the mixer with all the ingredients for about one and a half minutes. Superplasticizer was added for the batch where specimens were cured using added admixture. For the compressive strength tests, cylindrical specimen of diameter 150mm and height of 300mm were made according to the ASTM C 39. Fig 3.3 shows the casting of specimens. Test specimens were made as soon as practicable after mixing. The concrete was filled in to the moulds in layers approximately 50 mm deep using hand scoop. In placing each scoopful of concrete, the scoop was moved around the top edge of the mould as the concrete slides from it, in order to ensure a symmetrical distribution of the concrete within the mould.

Fig 3.3 Casting of specimens

For C1 to C4 specimens same batch of concrete was mixed each time. The difference in these specimens was the curing techniques employed. However, for C5 and C6 sikament-520 BA was added in the concrete mix in an amount of 1% to weight of cement in the mix. For C7 concrete, lightweight aggregates of 230 kg in weight were added in the mix as fine aggregate replacement and specimens were internally cured. Water cement ratio for this curing regime was kept at 0.36 given the intrinsic absorption capacity of lightweight aggregates. For C8 internally cured specimens, super absorbent polymers were added at a rate of 2g per kg of cement. Water cement ratio of was calculated as 0.33 for this regime. Lastly, C9 specimens had shrinkage reducing admixture added at 1% of the weight of cement in the mix.

Each layer of concrete batch was compacted by 25 strokes of tamping rod. After the last layer has been compacted with overflowing concrete, the surface was finished with trowel. By keep pressing the trowel, it was moved forward and backward to give additional compaction to the top layer concrete and the surface is also finished simultaneously. After finishing the specimens,

they were kept in moist air environment for 24 hours. One hundred and twenty specimens were prepared and after a period of 24 hours, specimens were demoulded and marked. Specimens were divided into six categories with each category having twenty specimens in them. For the concrete mix, medium workability of 100 mm was selected. The workability achieved in C1 to C5 was between 92-95 mm, C6 117 mm, C7 85 mm, C8 79 mm, and C9 109 mm. No cured (NC) specimens were placed open in atmosphere with no curing application on them. Water cured (WC) specimens were kept in water till testing at the appropriate ages. Liquid membrane-forming compound cured (CC) specimens were applied with Masterkure 107i after the demoulding. Burlap cured (BC) specimens were cured using hessian cloth wrapped around the specimens and watered three times a day. Liquid membrane-forming compound with added admixture (ACC) specimens were prepared with batch having Sikament 520 BA added at a rate of 1 % by weight of cement and then coating the sample with Masterkure 107i. Burlap cured specimens with added admixture (ABC) specimens were cured using hessian cloth. LWA specimens were internally cured using its water repository traits. SAP was also used at a rate of 2g per kg of cement for internal cured specimens and lastly, shrinkage reducing admixture (SRA) was used for internal curing of concrete specimens.

3.3.3 Testing procedure

The tests for compressive strength were performed on 3, 7, 14, 28 days cured samples, and an average of three samples was taken. At the appropriate age, the specimens were removed from water and surface water was wiped off. Immediately after finding the weight, the specimens have to be tested before they become dry.

Fig 3.4 Cracking pattern of NC and ACC specimens

Place the sample in the testing machine and applied load on permissible rate. However, proper capping was done prior to placing sample in the compression machine to ensure uniform

distribution of load on the face of sample. The maximum (crushing) load applied to the specimen was recorded and strength of sample was evaluated. Load was applied as specimen was carefully observed and application of load was stopped immediately after the development of visible crack in sample. Twenty specimens were tested for each curing technique. After the compressive strength of each technique, samples were development of microstructure. In addition to these tests, samples were collected from the cracked samples at the age of 3, 7, 14 and 28 days and were subjected to scanning electron microscopy (SEM) and quantitative analysis by energy-dispersive spectroscopy (EDS). The electron beam used for EDS analysis provides qualitative analysis to determine which elements are present, semi-quantitative analysis to identify the chemical compounds, quantitative analysis to determine what is the concentration of each element, and mapping the distribution and concentration of each element. The quantitative analysis depends on the mass concentration of element in the sample detected by X-rays. This SEM analysis was used to make comparison of microstructure development using various techniques. To further elaborate on quantitative analysis, table 4.1 shows mass percentage of two major and important constituents.

Fig 3.5 Sample preparation for SEM analysis

The measured peak intensities are the reflection of concentration of elements, which can be translated into their characteristics in the strength development of concrete. EDS depicts the quantity of various compounds in the samples. While undertaking SEM/EDS investigations, extensive specimen study was done at various locations with different resolutions using electron beam as per ASTM test standards E1508-12a and C1723, and representative area of interest for each specimen was selected for results and analysis.

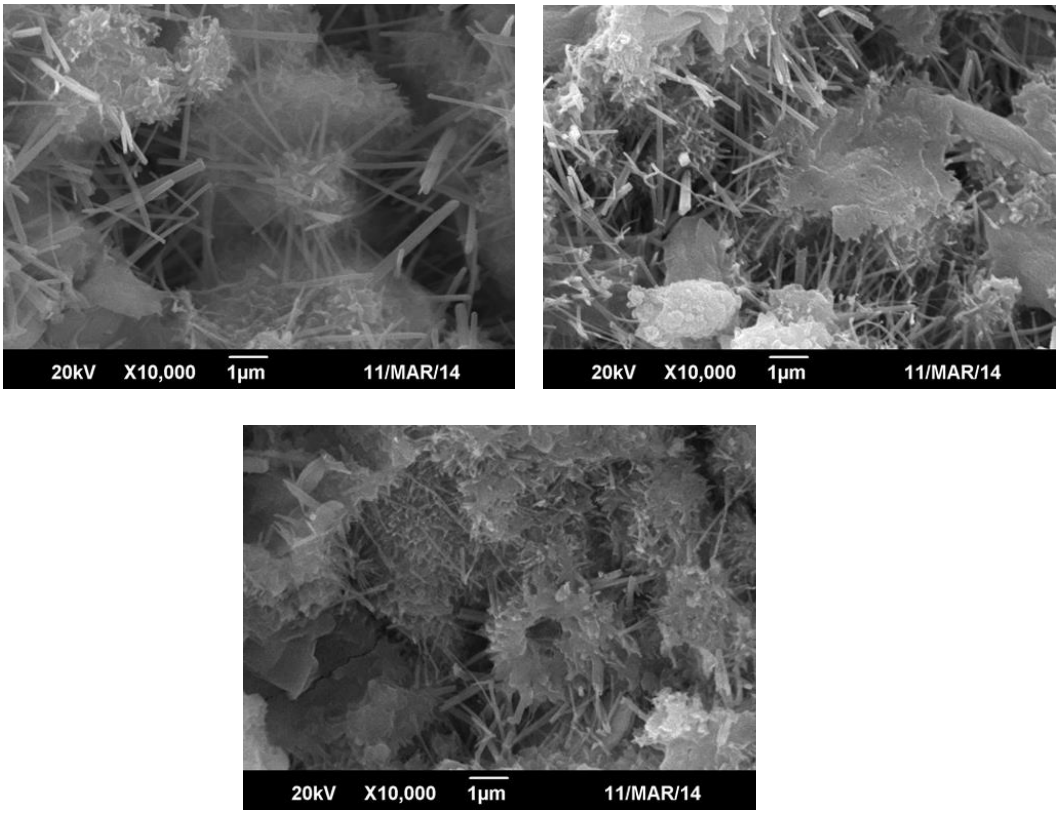


Fig 3.6 SEM analysis of NC, ABC and BC samples at 7 day testing at 10,000x magnification

Sample for SEM analysis requires complete moisture removal, for this purpose, quick coater machine is used. This machine completely removes the moisture from the specimen as well as coats the samples for SEM and EDS analysis. These samples, once completely dried, are subjected to SEM where the analysis is made at various magnification levels as shown in fig. 3.6.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 General

Results from previously mentioned tests are presented and discussed here to determine the efficiency of self-healing process of all mixes. These results include the crack width measurements, visual inspection of cracks, compressive strength of self-healing concrete samples and micro structural study through SEM and mineral composition of healing compounds through XRD analysis. Furthermore, relationships of self-healing at different curing days and compressive strength with self-healing techniques are elaborated.

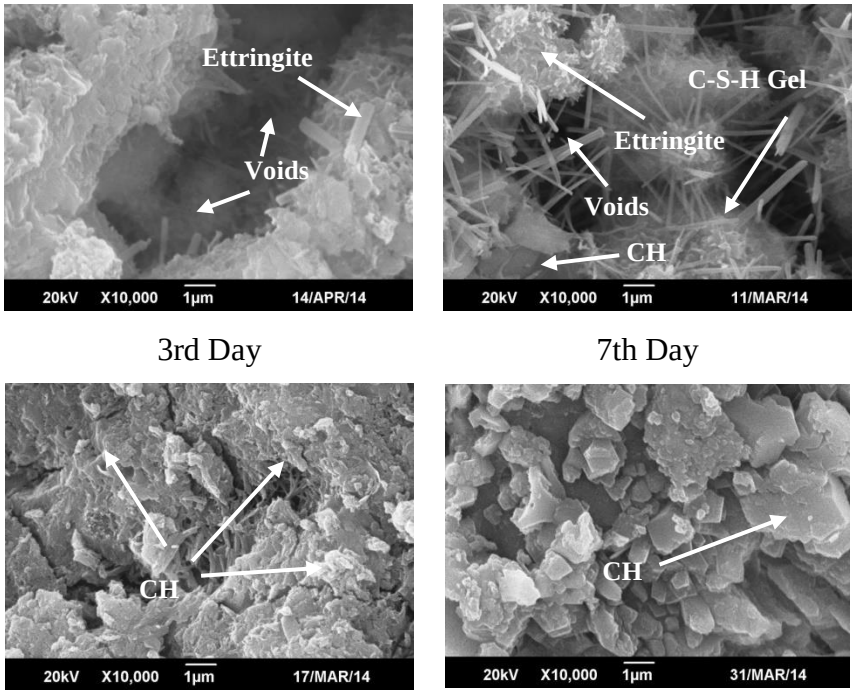
4.2 Microstructure and composition analysis

The results are presented to compare the effectiveness of various curing methods investigated. Figs. 4.1 through 4.18 illustrate SEM micrographs and EDS compositions for concrete specimens of various curing regimes designated as C1 through C9 at 3, 7, 14 and 28 days of casting respectively.

4.3 Air curing regime at ambient temperature

The SEM micrographs for C1 (air cured specimens at ambient conditions) in Fig. 4.1 indicate the formation of indistinct ettringite crystals on 3rd day. On 7th day testing, formation of C-S-H gel, CH plate like crystals and ettringite have clearly started to fill the empty spaces and help to improve the density. On 14th day, layered CH crystals are clearly seen with lesser proportion of C-S-H gel in it and on 28th day, similar situation is observed as of 14th day, with layered CH crystals existing in a very distinct formation. However, indistinct C-S-H gel formations are seen from the figure and honey-combs of C-S-H cannot be found in SEM analysis. The early strength in this regime is due to development of initial hydration products in concrete. However, as the hydration progresses the available water in the pores gets utilized by the constituents leaving empty pores behind. It is believed that the water in the capillary pores gets utilized reducing relative humidity of crystal lattice and as it reduces down to 80%, the hydration process stops hindering further development (Mindess et al., 2003). The 28th day EDS composition of C1 sample as represented in Fig. 4.2 shows Ca/Si (Calcium/Silicate) count ratio of 14.4 due to abnormality in the percentage of CaO, which shoots upto 700 counts. From table 4.1 it can be

clearly seen the development of unusual mass percentage of CaO leading to excess free lime distribution within the matrix and subsequent deterioration. The presence of free lime may be occurring due to unavailability of internal water that would have converted it into Ca(OH) (Raheem et al.) (Raheem et al.) (Raheem et al.) (Raheem et al.) (Raheem et al.) (Raheem et al.)$_2$ otherwise. CaO controls the strength and soundness in concrete, however, presence of excess CaO beyond certain value, makes it difficult to combine with other compounds and such availability of free lime causes unsoundness in concrete.



3rd Day 7th Day

14th Day 28th Day

Fig. 4.1 SEM micrographs of C1 (air cured) concrete at different ages

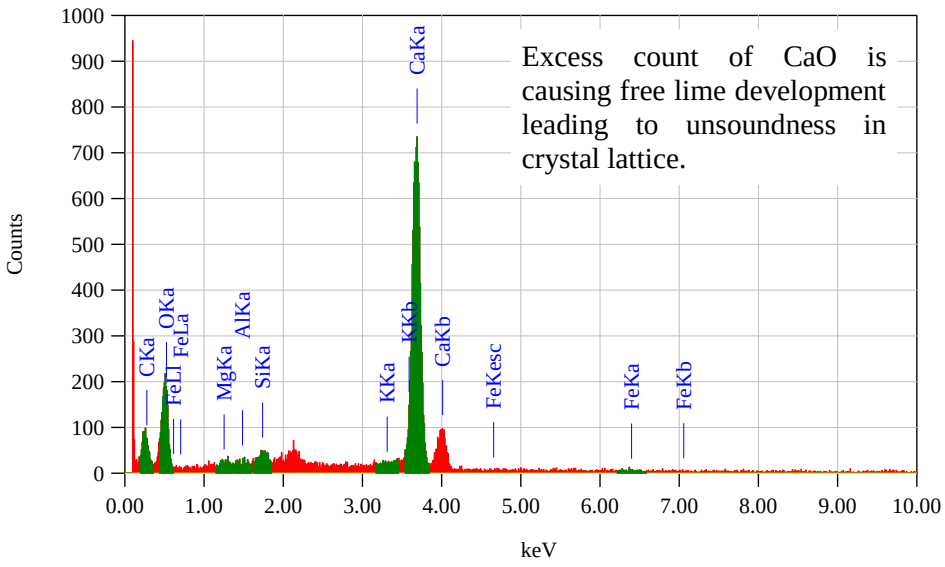
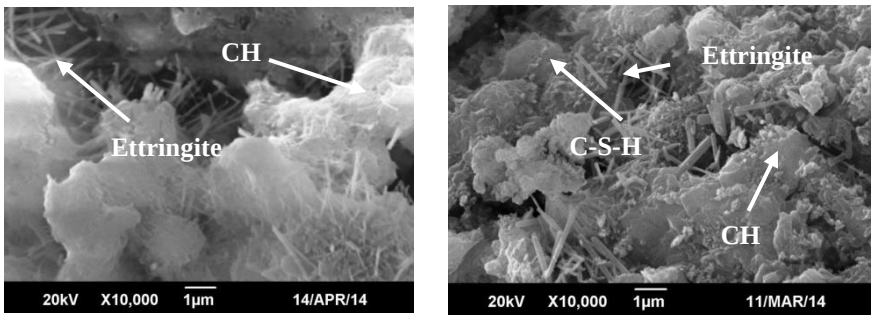


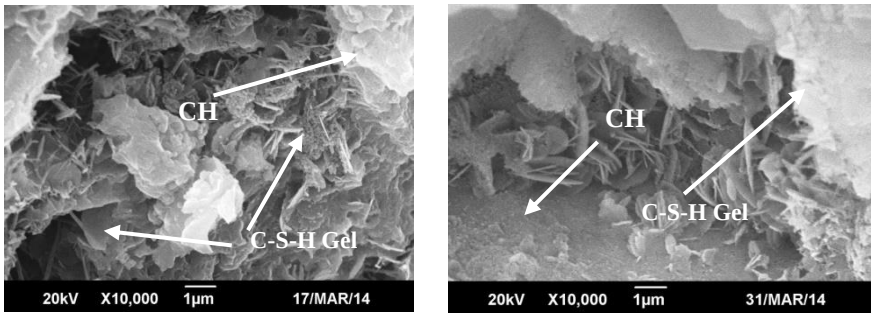
Fig. 4.2 EDS for C1 (air cured) concrete at different ages

4.4 Water immersed curing

The SEM micrograph for C2 (water immersed specimens) in Fig. 4.3 shows that C2 develops better microstructure owing the formation of hydration products with distinct ettringite and CH crystals on 3rd day testing. On 7th day, a clear formation of CH blocky plate like crystals and ettringite crystals in the voids can be seen. The honeycombs of C-S-H gel are also prominent indicating the hydration products resulting from proper curing. On 14th day, fine bundles of C-S-H (type – I C-S-H) and honeycombs of C-S-H gel (type – II C-S-H) are quite distinctive. 28th day testing illustrates a pure layered CH crystals with C-S-H gel identified by its honeycomb formation. Thereby, this techniques gives the best results among various other curing methods and conforms to previous studies (Kolo et al., 2013). EDS results in Fig. 4.4 show decrease in Ca/Si count ratio to as much as 11% of C1 indicating well-proportioned quantities of CaO and SiO_2 in the range of 61-67% and 15-24% respectively, conforming C_3S and C_2S in the acceptable ranges as stipulated in EN 197-1 specifications and contribute greatest in the strength development. Exact proportions of CaO and SiO_2 are important in imparting strength and soundness in the mixture.



3rd Day 7th Day



14th Day 28th Day

Fig. 4.3 SEM micrographs of C2 (water immersed) concrete at different ages

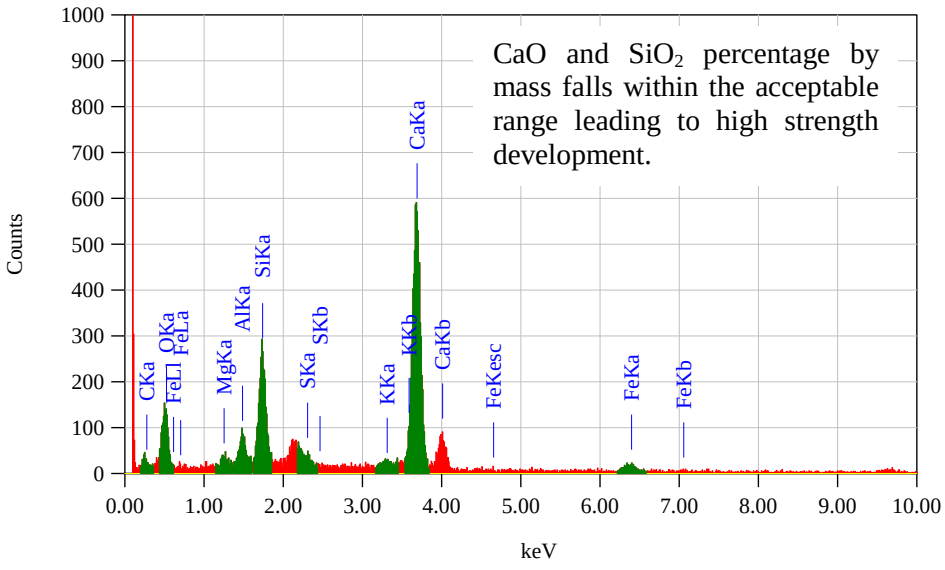
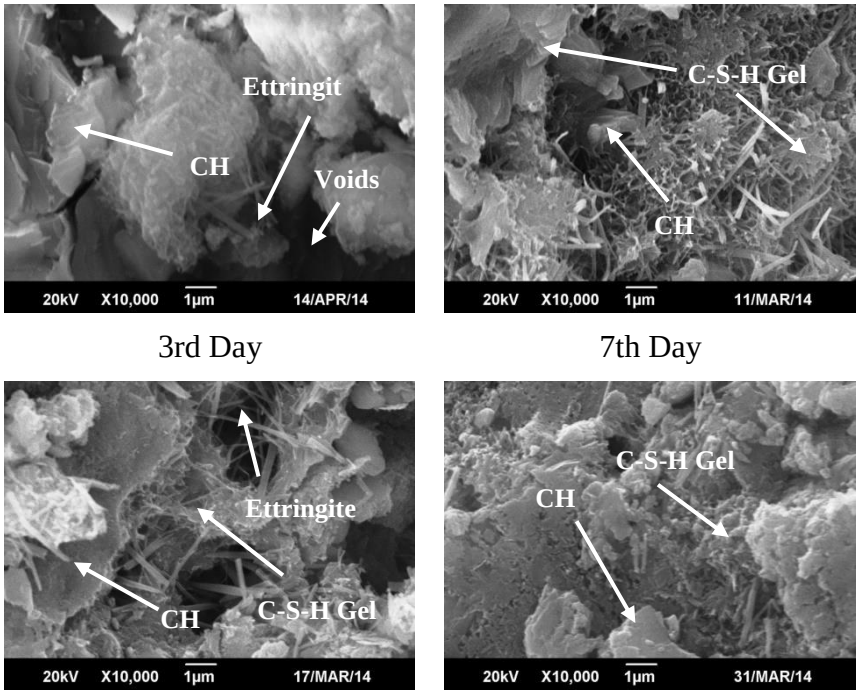


Fig. 4.4 EDS for C2 (water immersed) specimens at 28th day

4.5 Liquid membrane-forming compound curing

SEM micrograph in Fig. 4.5 for C3 (liquid membrane-forming compound cured specimens) illustrates an indistinct formation of ettringite at 3rd day testing while crystals of CH are seen in the formation phases.



3rd Day 7th Day

14th Day 28th Day

Fig. 4.5 SEM micrographs of C3 (liquid membrane-forming compound cured) concrete at different ages

On 7th day, honeycomb formation of C-S-H (type – II C-S-H) and platy-type CH are very distinct and substantially exist in the scanned area, which is different as compared to C1 and C2 curing. 14th day testing illustrates ettringite with its needle-like habitat, layered and blocky CH crystals and type-I C-S-H gel identified by honey comb and fine bundles formations similarly to identified by Franus et al. (2015). These C-S-H formations disappear on 28th day testing and replaced with platy-type C-S-H gel and CH crystals. EDS results of C3 in Fig. 4.6 show Ca/Si count ratio approximately equal to C2 while it is 13% of C1's count ratio representing oxides of calcium and silicon as 57% and 23.66% by mass respectively demonstrating acceptable ranges of primary oxides that lead to strength development.

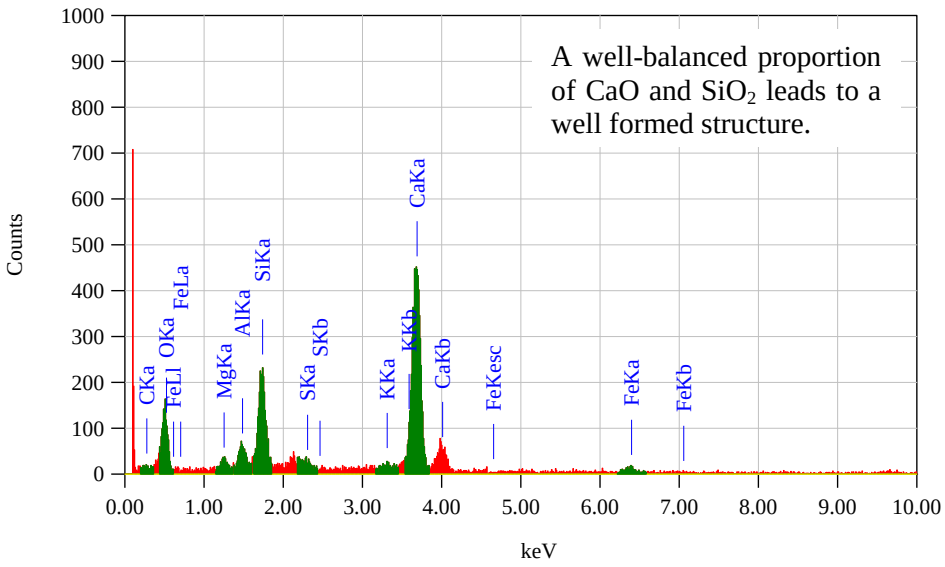
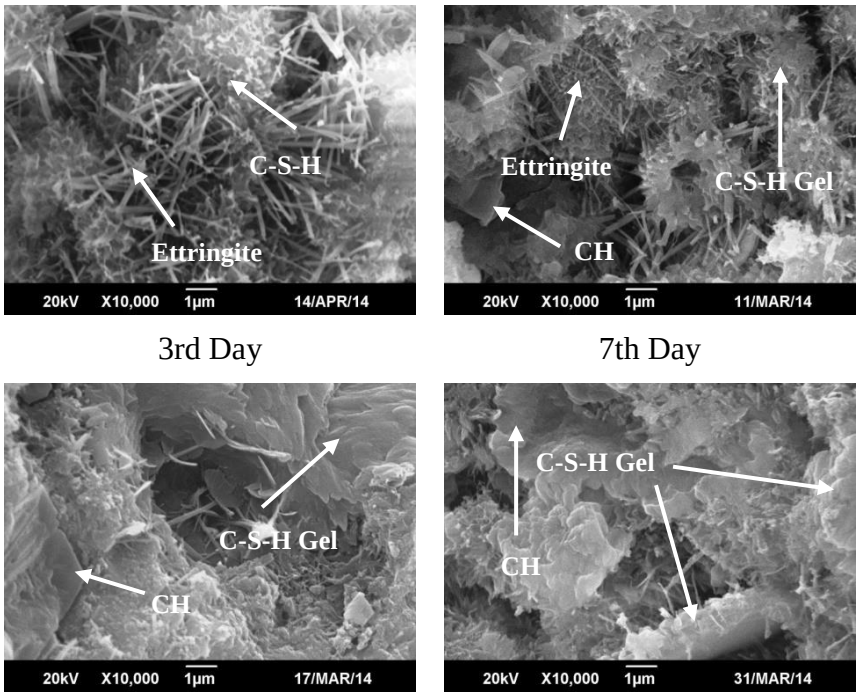


Fig. 4.6 EDS for C3 (liquid membrane-forming compound cured) specimens at 28th day

4.6 Wet burlap curing

Fig. 4.7 SEM micrograph shows C4 (wet burlap cured specimens) at 3, 7, 14 and 28th day. From the figures, it is clearly seen that on the 3rd day, formation of ettringite and honeycomb formations of C-S-H gel are distinct showing rapid development of microstructure. Similarly, 7th day micrograph illustrates a very discrete formation of CH platy crystals, ettringite needle-like structure and fine bundles and honey comb structure of C-S-H gel. On 14th day, fine honeycomb structure is mostly replaced by fine bundles like structure of C-S-H gel. On 28th day, formation of bundled structure along with honeycomb shaped C-S-H gel becomes prominent while CH layered and ettringite crystals seen indistinctly. This technique is suitable specifically in high temperature regions in the field with significant implications on the compressive strength of concrete (Gnana Venkatesh et al., 2014). In Fig. 4.8 EDS of C4 specimens has a Si/Ca count ratio reflected similar case as of C2 specimens with mass percentage of CaO and SiO_2 as 58.31% and 24.37% respectively as shown in table 4.1. The sum of proportions of CaO and SiO_2 in the mix should be not less than 50% by mass. This matrix fulfills the foregoing requirement and hence provides hospitable environment for the development of C-S-H gel structure and desired compressive strength of concrete.



3rd Day 7th Day

14th Day 28th Day

Fig. 4.7 SEM micrographs of C4 (wet burlap cured) concrete at different ages

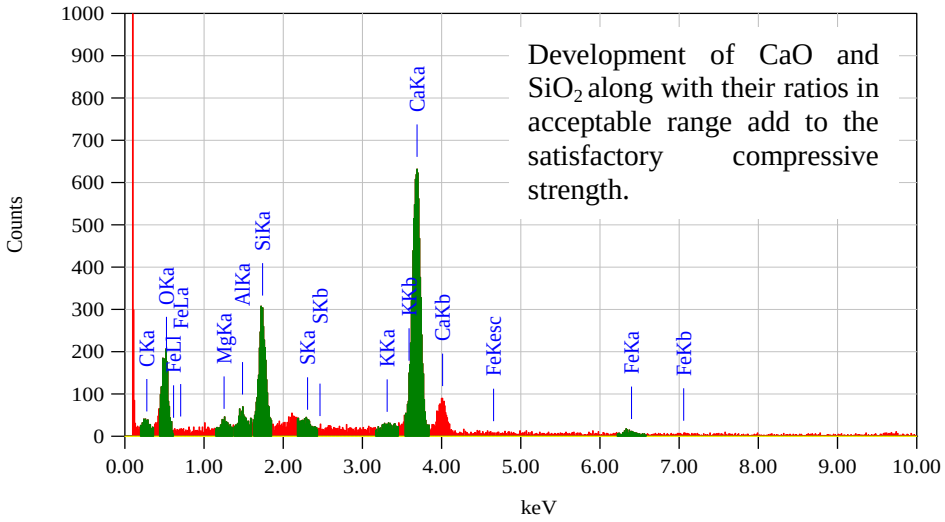
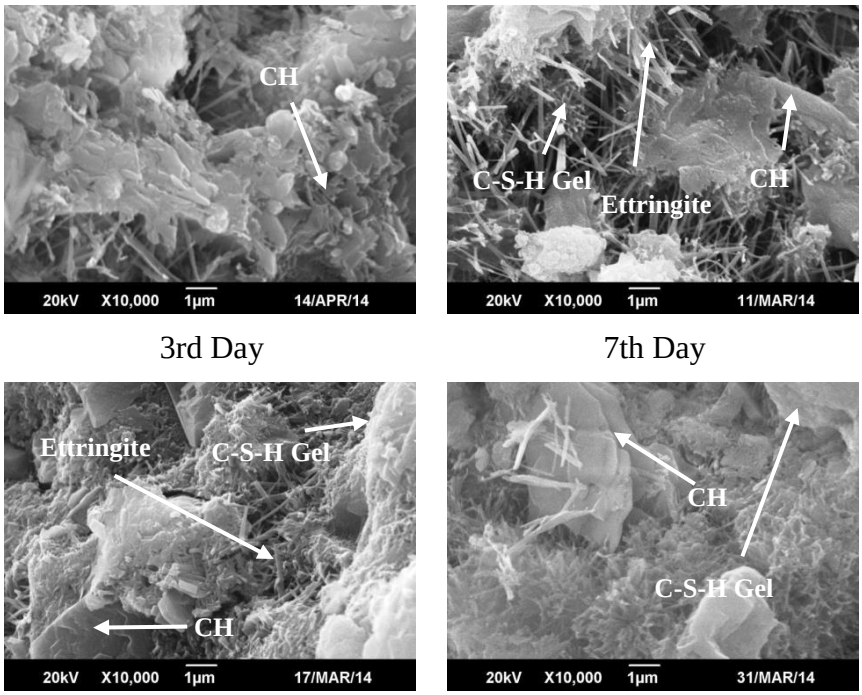


Fig. 4.8 EDS for C4 (wet burlap cured) specimens at 28th day

4.7 Admixture added liquid membrane-forming compound curing

In Fig. 4.9, C5 (admixture added liquid membrane-forming compound cured specimens) displays formation of CH crystals on 3rd day while few ettringite crystals are visible. On 7th day, formation of C-S-H bundles and honeycomb like structure is visible as well as development of CH crystal in shape of layered structure can be seen. Needlelike ettringite crystals can also be

observed in the micrograph. 14th day imaging illustrates matured microstructure of plate like CH, amorphous C-S-H and ettringite crystals while 28th day presents a more dense micro-structure, showing the positive effect of superplasticizer on the specified day (Franus et al., 2015). As in the case in C1, EDS of C5 in Fig. 4.10 specimens show Ca/Si count ratio increased about 200% of C2 and C4 indicating presence of free lime available in abundance causing expansion and disintegration of the specimens and consequently low compressive strength and unsoundness.



3rd Day 7th Day

14th Day 28th Day

Fig. 4.9 SEM micrographs of C5 (admixture added liquid membrane-forming compound cured) concrete at different ages

The mass concentration of both CaO and SiO2 as reflected by table 4.1 is 81.40% and 11.60% respectively. There are numerous admixtures and chemicals available commercially for the same purpose; however, type of curing material that presents favorable results together with chemical admixture may require careful consideration. It is therefore necessary that their effectiveness should be determined by making tests in the laboratory (Yilmaz and Turken, 2012).

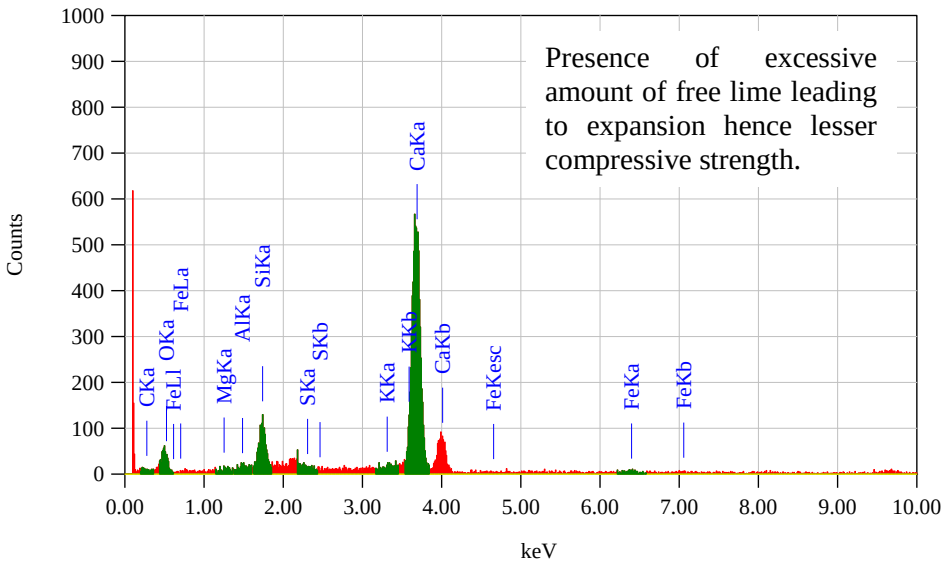
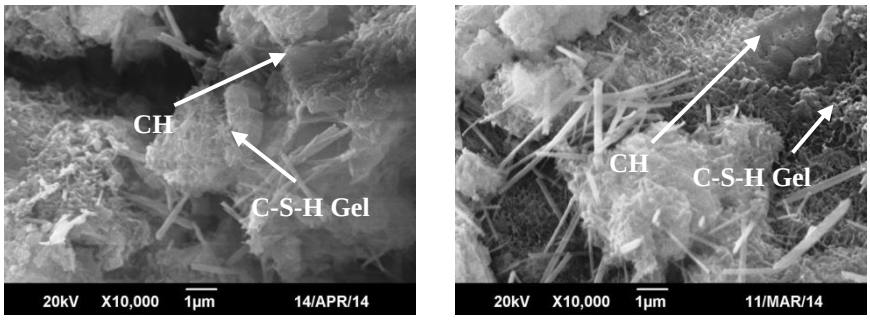


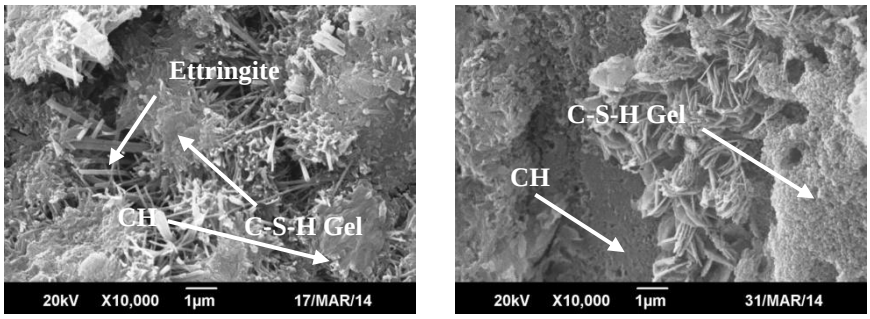
Fig. 4.10 EDS for C5 (admixture added liquid membrane-forming compound cured) specimens at 28th day

4.8 Admixture added burlap curing

Micrographs of C6 (admixture added burlap cured specimens) in Fig. 4.11 with 3rd day testing represent a different pattern as compared to C5 cured specimens as shown in Fig. 4.9. The images indicate the early formation of CH crystals and honey comb structure of C-S-H gel while ettringite is less to be seen. The 7th day image shows the development of CH crystals while the scattered C-S-H gel is under development phase indicating the slow hydration process. On 14th day, slow development is seen with partially hydrated structure of CH and fibrous C-S-H gel while unstable ettringite crystals decomposing into monosulfate hydrate are visible in this image (Mehta and Monteiro, 2006). The 28th day testing shows some of the fully hydrated particles of CH while scattered crystals of C-S-H are still partially developed at this stage. From the quantitative analysis for this technique in table 4.1, the obtained results showed higher mass percentage of SiO_2 as compared to other regimes reflecting the above mentioned phenomenon. The corresponding EDS of C6 specimens in Fig. 4.12 represents a Ca/Si count ratio 20% which is higher than C2 specimens showing excess of silica considerably more than other regimes in comparison to lime leading to slow setting of mixture.



14th Day 28th Day



14th Day 28th Day

Fig. 4.11 SEM micrographs of C6 (admixture added burlap cured) concrete at different ages

However, there is a satisfactory contribution of calcium and silicon oxides that eventually combine to develop a matrix that ensures meeting minimum requirement of compressive strength.

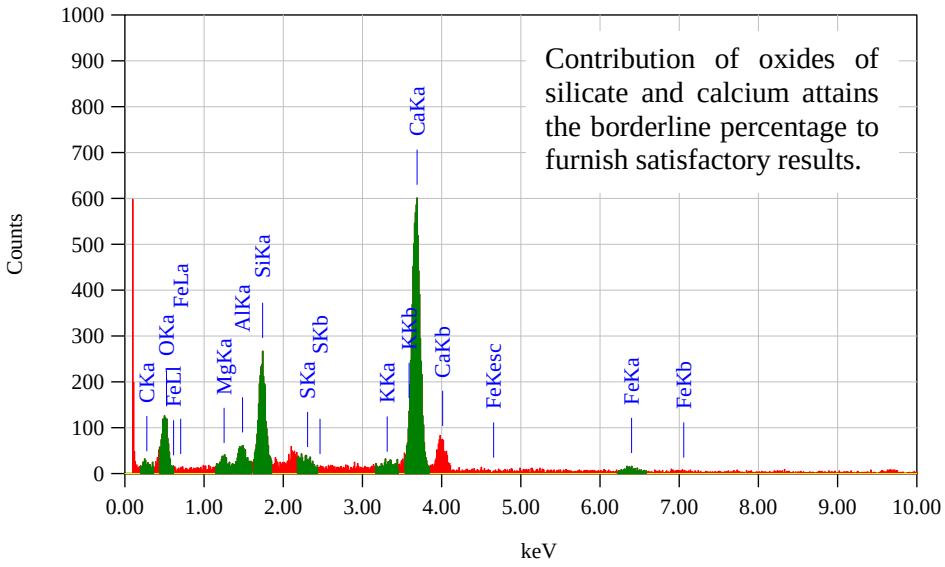


Fig. 4.12 EDS for C6 (admixture added burlap cured) specimens at 28th day

4.9 Lightweight aggregate internal curing

Micrographs of C7 samples with lightweight aggregates introduced as internal curing agent in Fig. 4.13 displays a stable hydration process which is attributed to its satisfactory strength at 28th day when compared with conventional curing regimes. A set of more distinctive fine bundles of type-I C-S-H and plate-like CH morphology are observed on 3rd day addressing its higher early compressive strength. On 7th day, compressive strength came out to be more than 70% indicating a saturation of platy-type-II C-S-H gel conforming to that proposed by Stutzman (2001) accumulating in layers in C7 specimens. Similar trend is observed on 14th day and 28th day with the structure at latter age showing an enhanced development of hydrated products leading to a higher compressive strength of more than 34.5 MPa. The successful use of LWA may also be attributed to its spatial distribution within the matrix which clearly demonstrates that internal drying of concrete pores induces a drying force by which water transports from pores of LWA into partially dried pores of cementitious system consequently increasing the compressive strength (Bentur et al., 2001). EDS in Fig. 4.14 firmly supports the excellent behavior of this curing regime with Ca/Si count ratio similarly to C2 showing upper ranges of calcium and silica present in the matrix that validates dense microstructure development.

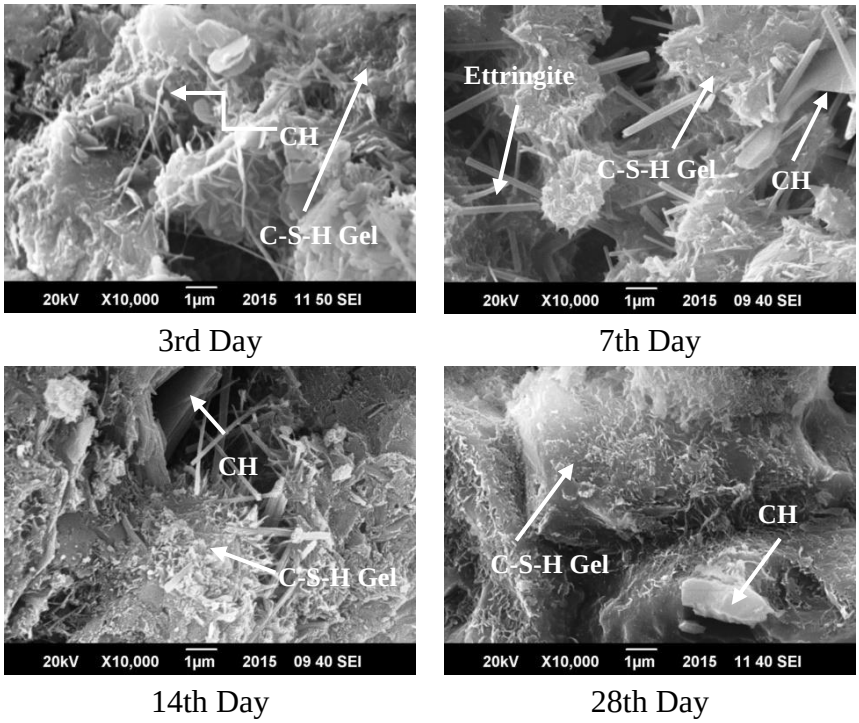


Fig. 4.13 SEM micrographs of C7 (lightweight aggregate) cured concrete at different ages

4.10 Super absorbent polymer internal curing

C8 specimens were administered using super absorbent polymers (SAP) as internal curing agent. As mentioned earlier, SAP showed an impressive however inconsistent behavior during the hydration process.

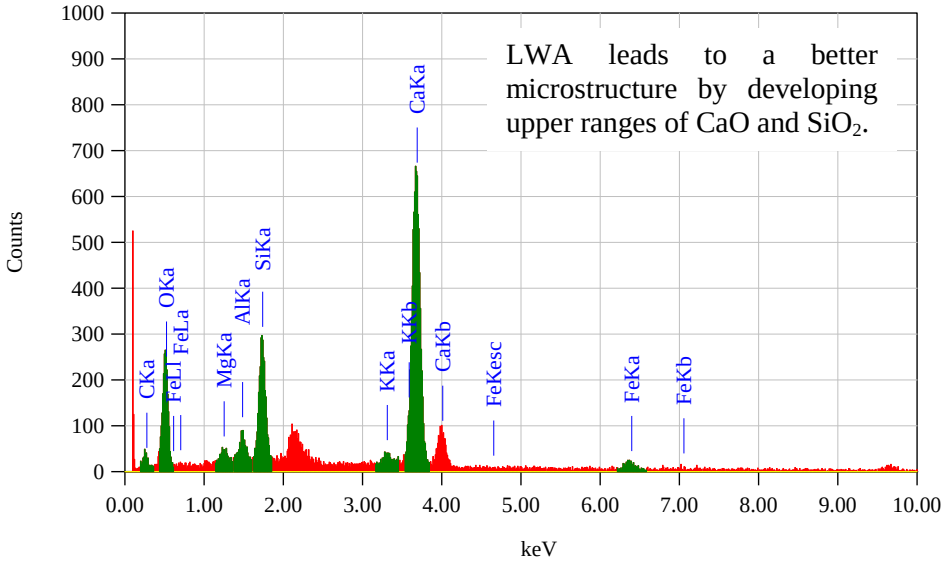
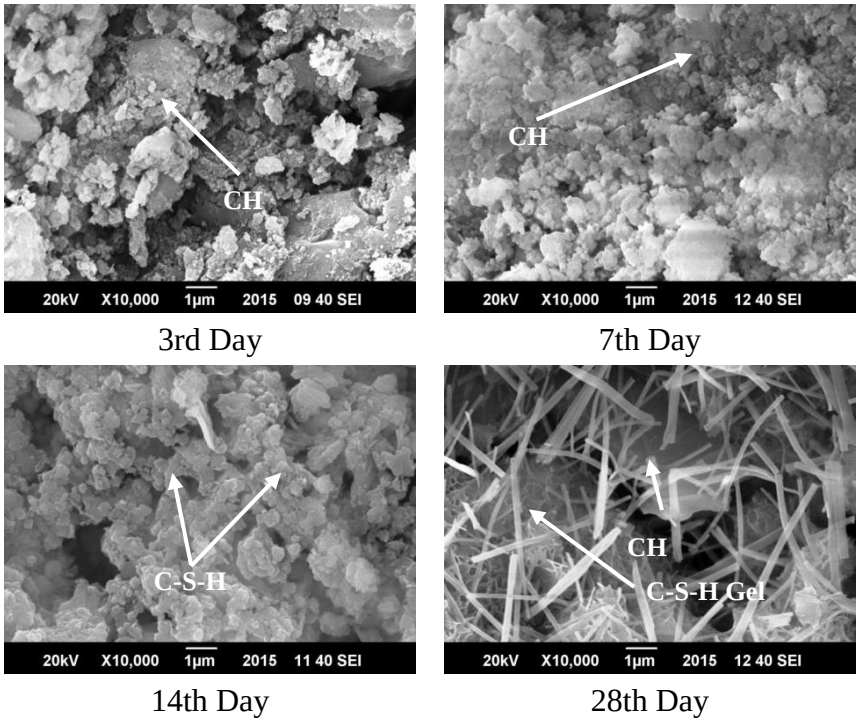


Fig. 4.14 EDS for C7 (lightweight aggregate) specimens at 28th day



3rd Day 7th Day

14th Day 28th Day

Fig. 4.15 SEM micrographs of C8 (super absorbent polymer) cured concrete at different ages

Fig. 4.15 shows presence of ettringite at 3rd day and 7th day was uncertain since the micrographs indicate a disarray of hydrated product in form of CH crystals which is a characteristic striated appearance as a consequence of crystal fracture within the paste on 14th day leading to lower compressive strength at early stages (Mindess et al., 2003). However, more elaborated picture was obtained at later stage along with desired results that favor SAP as internal curing agent.

During the early hydration, a low density arrangement of thin sheets of C-S-H gel was observed on 14th day which has higher porosity (Mindess et al., 2003). 28th day micrograph exhibited a dense microstructure; however voids can still be seen indicating a minor strength loss. An increased compressive strength due to application of SAP as internal curing agent was observed since it helps reduce the self-desiccation as observed by Klemm and Sikora (2011). SAP's EDS in Fig. 4.16 shows a 10% lower Ca/Si count ratio as compared to C2 at 28th day indicating a deficient lime presence in the structure, which leads to lesser strength. Table 4.1 shows the least mass percentage of CaO out of all the curing regimes with 44.26% and 26.39% of SiO2 at 28th day. However, significant filling of pores through C-S-H formation using internal water reservoirs with the support of microstructure development proved out to be satisfactory in terms of its targeted compressive strength.

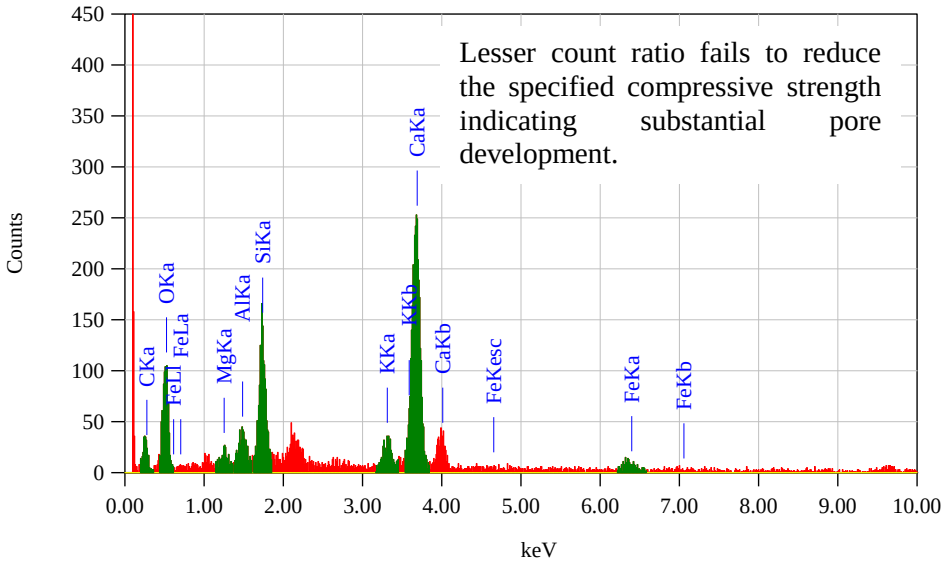


Fig. 4.16 EDS for C8 (super absorbent polymer) specimens at 28th day

4.11 Shrinkage reducing admixture internal curing

The micrographs of specimens containing shrinkage reducing admixture (SRA) in mix C9 are shown in Fig. 4.17. The curing technique did not firmly support the reason for application as both microstructure and compressive strength results implied an unfavorable resultant of hydration products.

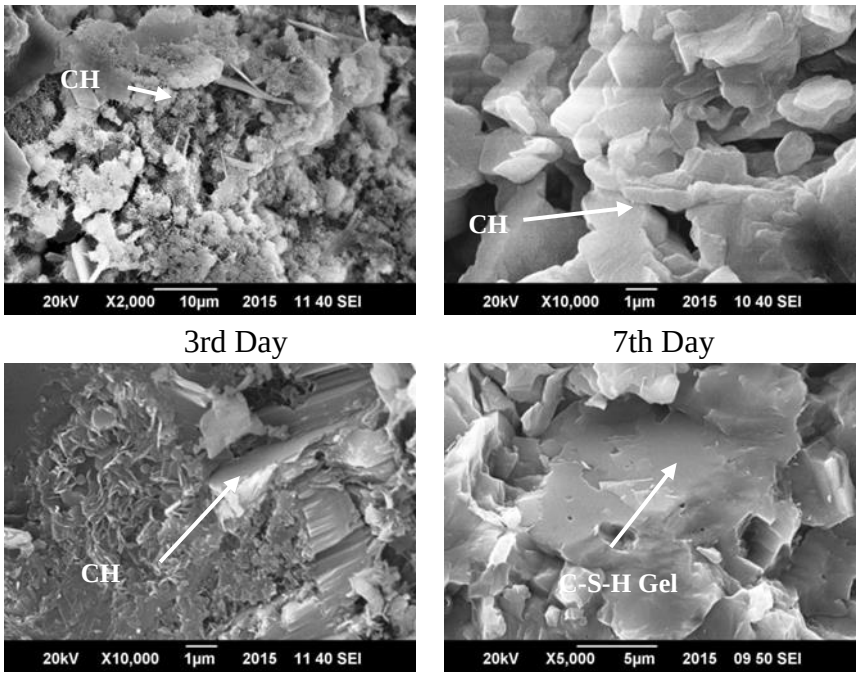


Fig. 4.17 SEM micrographs of C9 (shrinkage reducing admixture) cured concrete at different ages

The decrease in strength is believed to non-uniform distribution and underdeveloped hydrated products in the concrete (Mindess et al., 2003). At 3rd day, cactus shaped structure obtained with ettringite needles in an under developed form. The structure at consecutive testing ages showed similar products throughout, reflecting unsatisfactory and contrary outcomes of using SRA. Analogous to EDS of C1, SRA's EDS in Fig. 4.18 showed aberrant Ca/Si count ratio with 88.26% of CaO and just 3.53 % of SiO2 mass percentage as mentioned in table 4.1, showing excess of CaO presence throughout the system leading to expansion and disintegration of hydration products and unsoundness in concrete.

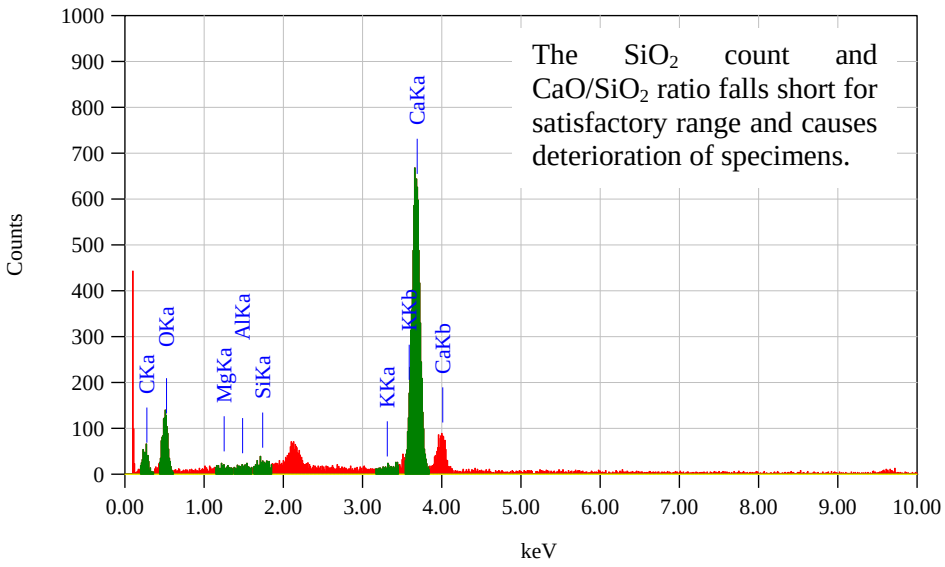


Fig. 4.18 EDS for C9 (shrinkage reducing admixture) specimens at 28th day

The microstructure and consequent compressive strength results complement each other based on the states of development at different ages. C1 specimens showed excellent growth of microstructure at the initial stage dominating the rest of the regimes however; it suffered a stalled condition after 14th day due to loss of internal water. C2, being the controlled specimens, showed anticipated results being most effective curing regime. C3 specimens showed development of microstructure at an optimum growth rate and consequently achieved 85% on field compressive strength requirement ACI 318-14 (2014). C4 and C6 specimens can be suitable for early development of strength as the density of microstructure grew substantially well with these curing regimes. In case of sealed curing, whether liquid membrane-forming compound cured or jute bag cured, it is experimentally found that concrete will keep on hydrating even if it is not fully saturated. This is because of the fact that water is held primarily in larger capillary pores held by surface tension of the concrete (Mindess et al., 2003). Cement draws water on these pores reducing relative humidity and thus slows down the rate of hydration. In a sealed environment, the rate of hydration and gain of strength is slow as compared to fully saturated concrete (Mindess et al., 2003). C7 specimens proved to be an indication for potential use of lightweight aggregate in future since it demonstrated an excellent microstructure development within the matrix and can be used without considerable detrimental effects when added to eliminate self-desiccation (Zhutovsky et al., 2004). C8 specimens with super absorbent polymers showed an erratic behavior however fulfilled the minimum compressive strength requirement

eventually. C5 and C9 curing regimes failed to develop the desired microstructure in the matrix leading to deterioration of specimens and decrease in compressive strength at various ages against their counterparts as illustrated through SEM and EDS analyses.

Table 4.1 - Elemental composition, masses and relative ratios of CaO and SiO_2

S/No	Curing Techniques		Mass (%)		Ratio
			CaO	SiO_2	Ca/SiO_2
1	Air curing	C1	86.34	5.32	17.2
2	Water curing	C2	57.28	24.41	2.38
3	Liquid membrane-forming compound curing	C3	57.14	23.66	2.42
4	Burlap curing	C4	58.31	24.37	2.39
5	Admixture added liquid membrane-forming compound curing	C5	81.40	11.69	6.96
6	Admixture added burlap curing	C6	59.70	29.45	2.03
7	Lightweight aggregate added internal curing	C7	57.21	22.78	2.51
8	Super absorbent polymer added internal curing	C8	44.26	26.39	1.68
9	Shrinkage reducing admixture addition effect on internal curing	C9	88.26	3.53	25

4.12 Compressive strength analysis

The compressive strength results for different curing techniques are shown in Fig. 4.19. It can be seen that water immersed specimens being the controlled specimens achieved the highest strength for 34.5 MPa specified compressive strength of concrete. Water cured cylinders resulted in better microstructure development and gave higher compressive strength (Qureshi et al., 2010) than sealed specimens conforming to explanations by Aitcin et al. (1994). For the 3rd day compressive test results, C1, C5 and C6 specimens showed almost same results while the compressive strength of C3 and C2 specimens showed substantially higher strength, with C2 specimens exhibiting the highest compressive strength results substantiating its well-known effectiveness. On 3rd day, formation of CH in C1 is very distinct which plays no role in the strength development of structure, while in C5 and C6 formation of CH crystals are more prominent which merely contain 15% of total area of products and do not contribute in the

strength development of structure. However, in C2, C3, and C4, development of C-S-H gel is more discreet which plays major role in the strength development and covers more than 50% of the hydrated matrix. On 7th day, C1 cured specimens showed substantially more strength than C3 specimens which is unusual since at this age formation C-S-H gel in C3 specimens is more defined whereas in C1 C-S-H gel is partially developed. This can be attributed to drying of specimens that decreases the volume of hardened cement paste caused by surface tension (Mindess et al., 2003). This surface tension increases in water-filled small pores, which results into secondary bonds between these surfaces thus increasing the strength (Popovics, 1986). Moreover, C1 micrographs' evolution from 3rd till 28th day shows abrupt changes indicating partially hydrated structure. Reason being that as chemically bound water is consumed in the hydration process no additional water is available to continue the hydration process, which results in collapse of CSH gel particles leaving large volume of porosity inside the system. This porosity therefore decreases the ultimate compressive strength of specimens.

In C5 and C6 specimens, formation of C-S-H gel is clear and prominent in the latter phase. After 14 days, C2 showed considerably high compressive strength with C4 specimens to follow, while C6 also resulted in increased compressive strength. C2 shows formation of C-S-H gel combined with CH resulting in dense microstructure. Same is the case with C4 specimens that shows the availability of water and its effectiveness in development of microstructure indicating a strength development more evident than other sealed curing specimens conforming to Al-Gahtani (2010) works. Same case is encountered on 14th day testing where C1 specimens achieved more strength than C3 specimens did which still has unhydrated particles due to rapid hydration process owing to unavailability of curing water and using internal water for development of structure. However, this rapid hydration for C1 resulted in unavailability of internal water and stopped further hydration consequently failed to meet the minimum specified compressive strength (Raheem et al., 2013). C6 specimens showed increasing strength development after 14th day because of the rapid development of CH crystals combined with C-S-H gel. After 28 days, C2 specimens as expected, showed the highest compressive strength while C4 specimens showed second highest compressive strength compared to other curing methods which corresponds to the earlier studies (Gnana Venkatesh et al., 2014).

Results obtained through application of internal curing medium were quite remarkable with LWA as water reservoir proved to be an efficient source of replenishing water demand inside the

system (Dayalan and Buellah, 2014). C7 samples showed a significant consistency in achieving satisfactory results after 28 days also seen in earlier investigations (Bentz and Stutzman, 2008). Compressive strength can be improved without having to reduce the effective w/c ratio by using LWA (Cusson and Hoogeveen, 2008). C8 specimens with SAP had inconsistent behavior at start however; it ended up with reasonably good results. It was found that degree of hydration of SAP aided internal cured specimens was higher after 14 days of testing. From SEM micrographs, it is apparent that as soon as SAP was added, it absorbed the available water inside the system leaving it hostile for hydration process as shown in 3rd and 7th day micrographs. At the outset of shrinkage, the water filled SAP started desorption process of releasing water upon pressure which resulted in further hydration process as indicated by 28th day SEM as well as compressive strength results. Although C8 did not achieve the target barrier compressive strength of 34 MPa and only shied away by 0.50 MPa, this technique is an indicator to practical implementation that shall prove to be a state-of-the-art technique for future endeavors. It was found that adequate use of SAP for low water cement ratios increases the compressive strength of concrete (Bentz and Weiss, 2011). C9 specimens could not be able to develop the desired microstructure that is responsible for the strength development and failed to fulfill the minimum specified compressive strength requirement.

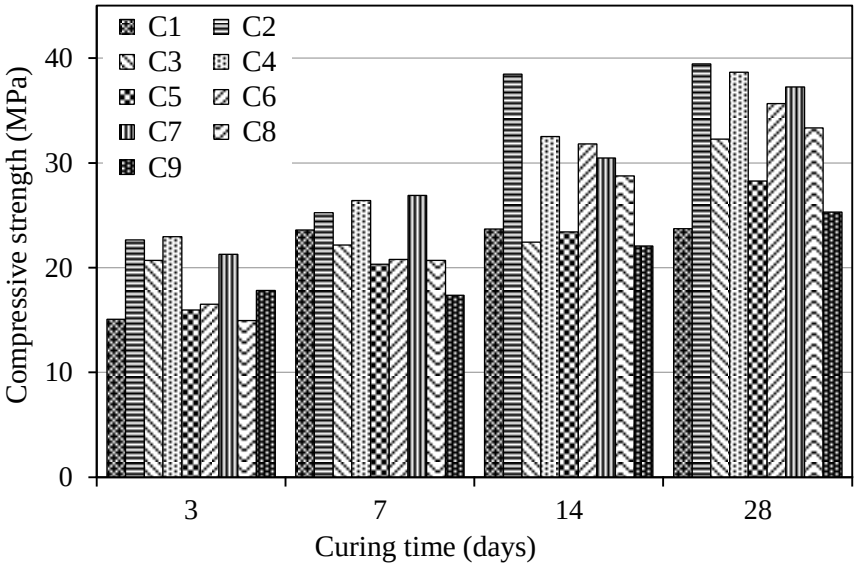


Fig. 4.19 Comparison of compressive strength development with time for different curing techniques

The results indicated that air cured specimens as C1 showed notable compressive strength development at start however it could not meet the minimum criteria of specified compressive strength at later stages. The highest compressive strength results were obtained in case of C2 specimens proving the effectiveness of water immersed curing. The developed strengths in C3 specimens indicate that liquid membrane-forming compounds may be used to for curing reinforced cement concrete without any negative effect on the plastic and drying shrinkage cracks and it implies positively for areas with scarcity of water or where availability of water is difficult (Qureshi et al., 2010). Higher compressive strength development was obtained in the C4 concrete specimens at 28 days than those cured by application of liquid membrane-forming compounds. Similarly, compressive strength obtained in C5 is less than C6 cured specimens, indicating higher strength and better microstructure development when superplasticizer is used with burlap curing (Al-Gahtani, 2010). Importance of internal curing was revealed through application of lightweight aggregates when on 7th day, C7 samples achieved same strength as conventional curing techniques without any external curing agent. C8 specimens made a slow recovery however fulfilled minimum specified compressive strength requirement at 28 days. According to ACI 318-14 (2014), compressive strength for 28-days cylinders, protected and cured to simulate good field practices, should test not less than about 85 percent of standard laboratory moist-cured cylinders. Results indicate that C3, C7 and C8 cured specimens have fulfilled the requirements with more than 90% of compressive strength which is higher than a requirement of 85% as stipulated in ACI 318-14 (2014).

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 General

The study aims to identify the efficient and effective method of curing that requires lesser time, has the tendency to develop better microstructure for durability and strength of concrete for regions where there are high ambient temperatures, high drying winds and low relative humidity. Permeability is a significant parameter which is directly associated with durability of concrete. The effect of development of microstructure on concrete durability performance is mandatory to establish the most efficient curing regime. Moreover, evaluating the most efficient curing regimes and associated durability performance can be most beneficially determined from the development of microstructure through various degree of hydration. According to ACI 318-14 (2014), compressive strength for 28-days specimens, protected and cured to simulate good field practice, should test not less than about 85 percent of standard laboratory moist-cured specimens. This rational basis needs to be validated using various conventional and emerging curing techniques.

5.2 Conclusions

The following conclusions are drawn based on the results obtained from various tests and discussion of findings.

- Initial compressive strength exhibited by air cured specimens by 7th day is noticeable, however it results into low ultimate compressive strength (70% of specified compressive strength), whereas water immersed curing helps attain the highest compressive strength.
- Liquid membrane-forming compound curing regime reduces the loss of moisture in concrete and helps development of dense microstructure.
- An improved microstructure development with lower shrinkage inclination of concrete reduced the micro cracking with LWA as internal water reservoir.
- Curing regime involving internal curing through LWA is helpful in provision of internal water repositories for uniform and continuous hydration, resulting in substantial reduction of self-desiccation. Concrete cured through incorporation of pre-wetted LWA therefore exhibits consistent and improved microstructural development leading to higher compressive strength and increased durability.

- In admixture added burlap cured specimens, slow growth of excess silica in the matrix affects early strength and microstructural development leading to lower initial age strength, however reasonably higher strength is attained at later ages.
- Internal curing through SAP exhibited slower strength development at initial stages; however, it was able to attain minimum specified compressive strength desired in field as per ACI.
- C5 (admixture added liquid membrane-forming compound cured) and C9 (shrinkage reducing admixture) curing regimes exhibited disarrayed microstructure and lowest compressive strength resulting from growth of excess free lime.

5.3 Recommendations

This research has provided significant understanding about concrete curing mechanisms and the effect of newly introduced internal curing techniques; further research is required to completely characterize the these mechanisms. Given below are some of the recommendations for further research in this area.

- Internal curing techniques are still incipient at research level in Pakistan, whereas it is non-existent at implementation level in construction industry. A research on larger scale should be conducted using readily available materials such as lightweight aggregate (LWA), clay or slate to make these techniques viable.
- Microstructure study through X-ray powder diffraction (XRD), X-ray fluorescence (XRF) and Backscattered electron (BSE) should be conducted using aforesaid additives to further elaborate the effects of hydrated products at various ages of concrete curing.
- A combination of internal curing materials may be utilized to evaluate their effects on the durability, shrinkage, and compressive strength of the concrete.
- Monitoring of variations in moisture contents/levels should be conducted during curing of concrete.

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