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Experimental Study on Strength and Durability Properties of Bio-Self-Cured Fly Ash Based Concrete under Aggressive Environments

R. Malathy

Abstract**—**High performance concrete is not only characterized by its high strength, workability, and durability but also by its smartness in performance without human care since the first day. If the concrete can cure on its own without external curing without compromising its strength and durability, then it is said to be high performance self-curing concrete. In this paper, an attempt is made on the performance study of internally cured concrete using biomaterials, namely *Spinacea pleracea* and *Calatropis gigantea* as self-curing agents, and it is compared with the performance of concrete with existing self-cure chemical, namely polyethylene glycol. The present paper focuses on workability, strength, and durability study on M20, M30, and M40 grade concretes replacing 30% of fly ash for cement. The optimum dosage of *Spinacea pleracea, Calatropis gigantea*, and polyethylene glycol was taken as 0.6%, 0.24%, and 0.3% by weight of cement from the earlier research studies. From the slump tests performed, it was found that there is a minimum variation between conventional concrete and self-cured concrete. The strength activity index is determined by keeping compressive strength of conventionally cured concrete for 28 days as unity and observed that, for self-cured concrete, it is more than 1 after 28 days and more than 1.15 after 56 days because of secondary reaction of fly ash. The performance study of concretes in aggressive environment like acid attack, sea water attack, and chloride attack was made, and the results are positive and encouraging in bio-self-cured concretes which are ecofriendly, cost effective, and high performance materials.

Keywords**—**Biomaterials, *Calatropis gigantea*, polyethylene glycol, *Spinacea oleracea*, self-curing concrete.

I. Introduction

HIGH performance concrete, falling into the category of the so-called modern concrete, is essentially characterized by a cement matrix with low water/cement ratio (w/c), often including mineral additions like silica fume and the use of admixtures as super plasticizers [1]. One of the major problems with these mixtures is their tendency to undergo early-age cracking. While this cracking may or may not compromise the compressive strength of these concretes, it likely does compromise their long-term durability. The phenomenon of early-age cracking is vulnerable and depends on thermal effects, autogenous strains and stresses, drying stress relaxation. Structural detailing and execution was dealt at length [2], [3]. Curing is the critical period of fresh cement in the hardening process, in which concrete develops its fundamental characteristics under specific conditions [4]. A proper curing essentially involves the permanent availability of internal moisture to sustain the hydration reactions at moderate temperatures, and in the absence of external forces at early ages. High performance concrete is particularly sensible to curing problems and may undergo considerable early-age deformations and microcracking development throughout the system [4], [5]. The dense microstructure of modern concrete rapidly develops capillary discontinuity in the still younger porous network. Thus, the access of external water turns out to be unviable to assure continuous saturation of the total bulk volume [6]. Concrete with internal curing may also have evolved from the concept of self-curing concrete, which is based on the introduction of a chemical admixture that is able to reduce water evaporation by a retaining function. The addition of a self-cure chemical based on a water-soluble polymeric glycol leads to improved durability of concrete cured in air [7]. However, the performance of such admixture does not attain the efficiency of the water film curing [8]. The use of water retaining agents should not be seen as internal curing, since it is conceptually based on the internal sealing rather than internal curing. The latter consists of a water-curing agent capable of enhancing cement hydration maintaining optimal curing conditions. Moreover, the selection of a specific curing method is highly dependent on actual design constraints [9]. In either case, there are many examples that require other form of curing procedures rather than the traditional or external curing methods, e.g. high strength concrete applied in submerged pipe systems within oil platform structures. In high performance concrete, capillary discontinuity may significantly delay and even limit the water movement throughout the system. This makes the use of water ponding or other external curing methods inefficient in assuring the continuous internal moisture of the material. In this case, the use of internal curing methods may be required to counteract autogenous deformation. As there is only little experience on the practical use of internal curing methods to improve early-age properties of high performance concrete, the subject constitutes per se a great challenge for technicians and industrialists of the modern age.

The need for internal curing initiates directly from the basic nature of cement hydration process. As a mixture of cement and water reacts to form crystalline and gel hydration products, the water combined into these hydration products generally occupies less space than water in its bulk form. So,

R. Malathy (Professor and Dean R&D) Department of Civil Engineering is with the Sona College of Technology, Salem, Tamilnadu, India (e-mail: malathycivil@sonatech.ac.in).

the hydration (and pozzolanic) reactions are accompanied by a net chemical shrinkage as the products occupy less space than the reactants. The hydration of tricalcium silicate reduces about 9.6% by its volume, in the other words, about 0.07 mL/g by considering molar volumes in reaction with 7% on a mass of water needed to mass of C_3S basis [10]. This chemical shrinkage will lead to physical shrinkage of three-dimensional microstructure of cement, before it sets [11], [12]. However, after the cement paste sets and develops a finite resistance to deformation, the chemical shrinkage, in the absence of additional water, may lead to self-desiccation, and pores with partial satisfying may be formed within the microstructure [13]. The pore solution menisci remaining in partially filled pores will create a quantifiable capillary pressure, directly relative to the surface tension of the pore solution and inversely proportional to the size of the largest partially filled pore.

Young's equation as developed by Alberty and Daniels [14] relationship between capillary pressure σ and surface tension of pore solution γ as $\sigma = (-2\gamma cos\theta)/r$, where θ and r are the contact angle and the pore radius, respectively. From Young's equation, it is clear that, for reducing the capillary pressure, the surface tension needs to be reduced, or the size of the pores has to be increased [1], [2]. In the absence of sacrificial larger pores, the capillary stresses will promptly rise over time, as minor and minor pores within the hydrating cement paste unfilled, whereas the ongoing hydration further reduces the size of the remaining water-filled capillary pores. The reduction in microstructure caused by capillary pressure can be estimated using Mackenzie's modified equation [15]. Early-age cracking caused by the autogenous stresses and strains will create open pathways for ingress of deleterious species. As per the equation given by Kelvin [14], increase in stress occurs due to self – desiccation, and consequently, there will be a reduction in the internal relative humidity of the hydrating cement paste. Hence, to keep the capillary porosity of the hydrating cement paste remaining saturated, a source of available additional water can be provided by the way of internal curing so as to minimize the autogenous stresses and strains. The available additional water will also help to maximize the hydration of cement and mineral admixtures in concrete.

II. Research Significance

The prevention of loss of moisture from concrete is important for the strength development. It is used for the prevention of plastic shrinkage, reduction in permeability, and improvement of resistance to abrasion. The loss in strength after 28 days seems to be directly related to loss of moisture during the first three days. So, internal curing is used as a substitute to overcome such problems. One of the methods to eliminate such problems faced by external curing is the use of lightweight aggregate. The other methods are the use of water absorbent polymers (polyethylene glycol and paraffin wax) and wood derived materials. The disadvantage of using such materials is that, being lightweight aggregate, it can negatively impact strength and can lead to variability in performance. Polyethylene glycols are more controllable but are relatively expensive compared to lightweight aggregate. Wood resultant materials may be substituted with other internal curing materials. It provides consistency at a lower cost, but only 50% strength is reached, and microcracks are introduced by wood derived materials. The above deficiencies can be overcome with bio-materials like *Calatropis gigantea* and *Spinacea oleracia* as curing agents without compromising the strength and durability properties of conventional concrete [16]-[19]. There is significant improvement in strength of fly ash based concretes that are internally cured with these biomaterials [20]. This paper made an attempt on studying the effect of those biomaterials for durability properties of fly ash based high performance concrete.

III. Materials and Mix Proportioning

A. Materials

Ordinary Portland cement OPC 43 Grade confirming to IS: 269-1976 was used throughout the investigation. Class F fly ash from thermal power plant is used in this study. It is light gray in colour, and its specific gravity is 2. Locally available blue granite metal was used for the preparation of concrete. Locally available hard blue granite metal, well graded 20 mm and down size were used. River sand passing through 4.75 mm sieve as per IS383 specifications confirming to zone II were used as fine aggregates. Table I depicts the physical properties of fine and coarse aggregate.

TABLE I
Physical Properties of Fine and Coarse Aggregate

Description	Fine aggregate	Coarse aggregate
Specific gravity	2.6	2.7
Water absorption	1.57%	2.3%
Fineness modulus	3.1(zone II)	6.4
Surface moisture	Nil	Nil
Bulk density	1450 kg/m^3	1750 kg/m^3

B. Self-Curing Agents

The test results concluded that, compared to conventional concrete, concrete with other curing agents, specifically with polyethylene glycol, gave better results. Having arrived at better results with *Spinacia oleracea* and *Calotropis gigantea*, the two curing agents were used in different concrete mix proportions with different quantity of curing agents and tested for compressive strength [16]-[20].

Existing curing agent available in the market is polyethylene glycol, and its molecular weight is between 190 and 210. The specific gravity is 1.12-1.13. Hydroxyl value is 535-590 (mg KOH/g), and pH value is between 5 and 7. *Spinacia oleracea* is usually called as Palak greens in Tamil Nadu and it is a type of green popularly consumed as food product. Curing agent was prepared from the filtrate extract of *Spinacia oleracea* after it was ground well. Its pH value is 6.59. This extract base is added at the time of preparing concrete that is while adding water to the dry ingredients. Placing and compacting the fresh concrete is similar to the conventional concrete, but without curing. The chemical structure shows that it contains (-O-) and (-OH) functional groups. As such, the *Spinacia oleracea* selected as internal curing agent possesses hydroxyl and ether functional group, which is also revealed in Fourier Transform Infra-Red (FTIR) results [20]. *Calotropis gigantea* is a waste plant which grows in fields and terrains without any special care or water. This milk is tried also as another curing agent. Its pH value is found to be 5.17, which is in the range of polyethylene glycol. The method of preparing the extract from the spinach leaves and the collection of milk oozing out of the *Calotropis gigantea* are shown in Figs. 1 (a) and (b).

Fig. 1 (a) Extraction of *Spinaica oleracea*

Fig. 1 (b) Milk from *Calotropis gigantea*

C Mix Proportioning

The mix design for concrete M20, M30, and M40 grades is arrived based on the code IS 10262:2009. The cement is replaced with 30% of fly ash by its weight. The dosage of curing agents was optimized and found to be *Spinacia oleracea* at 0.6% by weight of cement and fly ash, *Calotropis gigantea* at 0.24% by weight of cement and fly ash and polyethylene glycol at 0.3% by weight of cement and fly ash, and strength studies showed encouraging results [16]-[20]. Hence, those proportions were used for making fly ash based concrete samples for durability studies to evaluate long term property of those biomaterials compared with polyethylene glycol.

IV. Experimental Programme

The slump flow tests for workability and compressive strength test as per Indian standards were conducted. The durability properties under aggressive environments like acid attack, alkaline environment, abrasion, and chloride attack were studied.

The acid resistance tests were carried out on cube specimens immersed in water diluted with 1% by weight of sulphuric acid for 45 days continuously. Then, the weight and the compressive strength of the specimens were determined, and the average percentage of loss of weight and the percentage loss of compressive strengths were calculated.

The alkaline resistance tests were carried out on cube specimens which were weighed and immersed in water diluted with 3% sodium hydroxide by weight of water for 90 days continuously. Then, the compressive strength of the specimens was measured, and the average percentages of loss of compressive strengths were calculated.

The Rapid Chloride Ion Penetration test (RCPT) was performed as per ASTM C 1202 to determine the electrical conductance of M 20, M 30, and M 40 grades of mixes at the age of 28 days and to provide a rapid indication of its resistance to the penetration of chloride ions.

V. Results and Discussions

A. Workability

The slump values are shown in Fig. 2, and it is observed that the workability of concrete mixes for all the combinations of the internal curing agents is 4 to 8% lower than the conventionally cured concrete. Even though the slump is less for self-cured concrete when compared to conventionally cured concrete, it is found that concrete with all the curing agents (*Spinacia oleracea*, polyethylene glycol, and *Calotropis gigantea*) satisfies the standard of minimum slump of 50 mm. The slump loss in self cured concrete may be due to the absorption of water, and holding capacity of self-curing agents also may be the reason that total w/b ratio is maintained including the liquid content present in self curing agents.

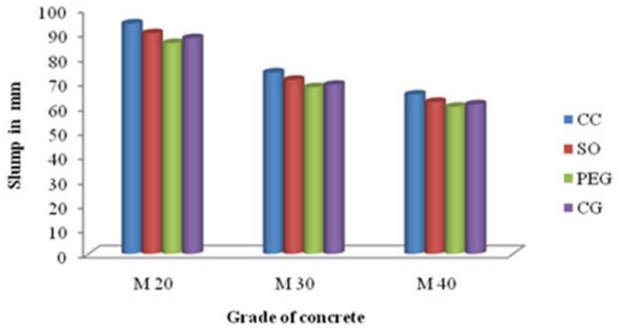


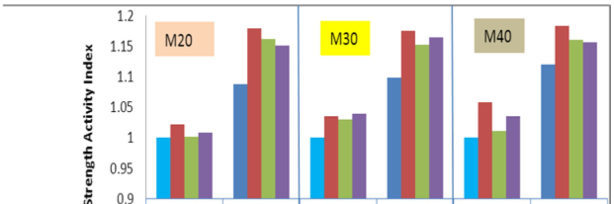
Fig. 2 Slump in mm for various grades of concrete

B. Compressive Strength

The strength activity index is calculated for internally cured concrete keeping unity for compressive strength of conventionally cured concrete at 28 days and it is shown in Fig. 3. The increase in strength at 56 days varies between 8 to 9% higher than that of strength at 28 days for different grades of conventionally cured concrete, whereas the increase in strength found in self-cured concrete is up to 18% because strength contribution by the fly ash is at later ages due to secondary pozzolanic reaction. The presence of ether and hydroxyl functional groups in the concrete enhances the formation of a continuous system of gel which provides better strength development at early ages. Continuous hydration of the mixture at later ages, promoted by the available water due to curing agent, also contributes to the strength increase. Microcracks due to aggregate restraint were minimized since cement paste expanded at early age rather than shrinks and hence improves the strength of concrete.

C. Durability

The results of the acid resistance tests of various concrete mixes at the age of 45 days were observed, and the percentage loss in weight and strength appears in Figs. 4 and 5. Thus, from the test results, it is observed that the attack of acid on concrete is less for concrete cubes with *Spinacia oleracea*. The percentage loss in weight in cubes with *Spinacia oleracea* is around 15% less when compared to conventional concrete and is around 10% and 5% in cubes with polyethylene glycol and *Calotropis gigantea*. Even though the water absorption and porosity values are higher for concrete with self-curing agent, namely *Spinacia oleracea*, the percentage loss in weight and compressive strength due to acid attack is lower. This is due to the fact that the percentage chance for the attack of concrete by acid is less as the pore structure of concrete becomes water tight and does not allow further entry of acid water into the concrete.



	M20 28 days	M20 56 days	M30 28 days	M30 56 days	M40 28 days	M40 56 days
Conventional concrete	1	1.088	1	1.098	1	1.12
Spinacia oleracea	1.022	1.18	1.036	1.175	1.058	1.184
Calotropis gigantea	1.002	1.162	1.03	1.153	1.011	1.161
Polyethelene Glycol	1.008	1.151	1.04	1.164	1.036	1.156

Fig. 3 Relative compressive strength for various grades of concrete

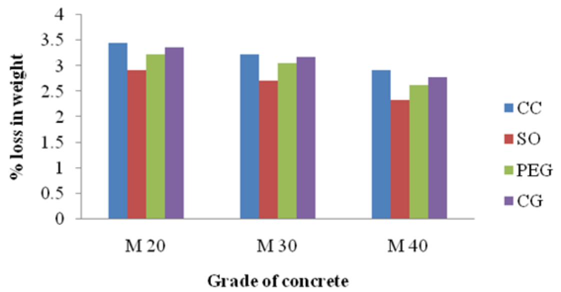


Fig. 4 Percentage loss in weight due to acid attack

Fig. 6 shows the loss of strength due to alkaline attack, and from the figure, it is observed that the concrete with *Spinacia oleracea* curing agent is more resistible against alkaline attack. The loss of strength in cubes with *Spinacia oleracea* is 20% to 30% lower compared to that of conventional concrete. The resistance in *Spinacia oleracea* is higher because further entry of alkaline liquid is prevented, whereas the loss of strength in cubes with polyethylene glycol is around 15% lower compared to conventional concrete. In cubes with *Calotropis gigantea*, the loss of strength is around 30%.

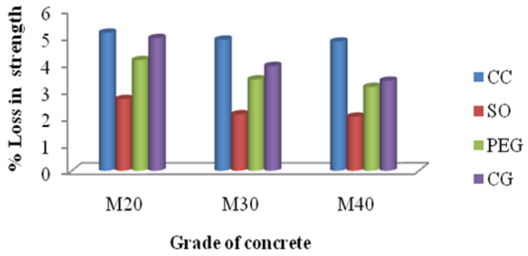


Fig. 5 Percentage loss in strength due to acid attack

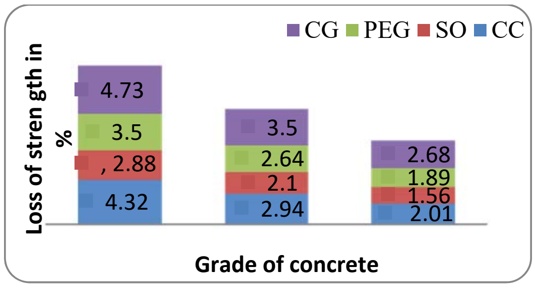


Fig. 6 Alkaline resistance for different grades of concrete

The abrasion resistance test results of various grades and types of concrete at the age of 28 days were observed. Abrasion resistance for different grades of concrete with different curing agents is expressed in percentage wear as shown in Fig. 7.

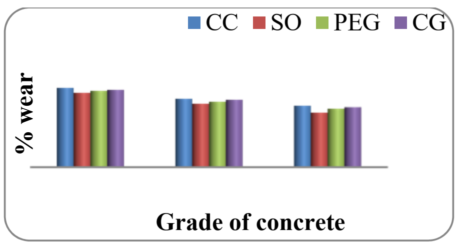


Fig. 7 Abrasion resistance for different grades of concrete

Chloride ion penetration in M 20, M 30, and M 40 grades of concrete for different curing agents is shown in Table II. All the values come under the low and moderate category. When the grade of concrete is higher, there is reduction in the magnitude of charge passed. The reduction in the magnitude of charge passed could be due to the depletion of calcium ions in the gel pore fluids and subsequent reduction of the pH and the development of constricted discontinuous and tortuous pore structure. The quantity of current flowing under the influence of voltage has a direct bearing on the characteristics of the pore solution since the diffusion of ions through the hardened concrete is only due to the electrolytic conduction.

As per ASTM C 1202, the conventional concrete of M 20 and M 30 grade falls under the category of 'Medium' degree of chloride penetrability. The rest of all the other samples falls under the 'Low' degree of chloride penetrability. Fig. 8 shows the specimens, after testing RCPT. Thus, the results of rapid chloride ion penetration test have demonstrated the superior durability characteristics of the mix.

Fig. 8 Specimens subjected to chloride penetration

TABLE II
RAPID CHLORIDE PENETRATION TEST AT THE AGE OF 28 DAYS

Concrete with curing agent	**Charge passed as per ASTM equivalent (Coulombs)**		
	M20	**M30**	**M40**
Conventionally cured concrete	2560	2400	1800
Concrete with *Spinacia oleracea*	1800	1350	1100
Concrete with polyethylene	1600	1440	1050
Concrete with *Calotropis gigantea*	1300	1250	1200

VI. CONCLUSION

From the workability, strength, and durability studies, it is revealed that there is a presence of OH ions in the self-curing concrete. This helps in the effective hydration resulting in better durability properties. It is concluded that the vegetative materials added as internal curing agents perform better workability, strength, and durability characteristics in fly ash based concrete of grades M20, M30 and M40, and such biomaterials as internal curing agents can be used for RCC works, pavements, water tanks, pre-stressed concrete structures without curing with fly ash to achieve long term strength with high performance.

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R, Malathy graduated in the year 1992, obtained M.E. (Structural Engineering) in 1998 both from Madras University, Chennai and Ph.D in 2005 from Bharathiyar University, Coimbatore. She has more than 25 years of teaching experience and 11 years in research. She is the principal investigator for 6 R&D projects (funded by DST, AICTE, IEI , UGC etc.,) worth more than 60 lakhs. She has done extensive research work on High Performance Concrete, Self Compacting Concrete, Self Curing Concrete, Geo Polymer Concrete, Papercrete and Effect of mineral and chemical admixtures on properties of Concrete. She published about 100 papers in reputed journals and conferences at international and National levels. Her recent researches are utilization of steel slag in concrete for various applications like pavements, precast prestressed elements and RCC elements. For her innovative research work on utilization of steel slag, JSW Steels, Macheri, honoured with an award on 15th August 2016. She is a member of various professional bodies such as IEI, ISTE, ICI, ACI (India chapter) and TNSF. Now she is the Chairman of Indian Concrete Institute ICI – Salem Centre and GC member of ICI. She received Best Teacher Award for the year 2004 in Kongu Engineering College and Innovative Teacher Award for the year 2005 from CLHRD, Mangalore. She got "Career Award for young Teachers" for the year 2006 from AICTE, New

Delhi. She also received "Best Paper Award" in the year 2006 published in ICI journal. She patented a product "Electronic Level Indicator using Sensors" in the year 2006 and now 3 patents are in 2011. She organized several workshops and conferences and delivered lectures on Concrete Technology, Effective Teaching Learning Process, Human Resource Management and Earthquake Engineering in various institutions and to practicing Engineers. She visited Singapore to present a paper on "Efficiency factor for silica fume and metakaloine" in International Conference on Our World in Concrete and also chaired one session. She visited South Korea on 24th September to present a paper on Geo Polymer Concrete in the 12th International Congress on Polymers in Concrete. She also visited London to present papers in International Conference and she has been invited as a resource person in the Conference on Building Materials in Germany. She received Best Engineering College Teacher Award from ISTE (TNC) and Best Women Engineer Award from Institution of Engineers in the year 2008. She has been recognized for inclusion as a biographee in the 2009 edition of Marquis' Who's Who in the World, a globally distributed publication continues to be recognized internationally as the premier biographical data source of notable living individuals from every significant field of endeavor. Under her guidance, Project students received Socio Engineer Award for the project regarding "treatment of dyeing factory effluent by using fly ash as an adsorbent". She received Best Senior Engineer award 2011 from Salem Chapter of IEI. She received two National awards from ISTE one is for her outstanding research work on development of eco friendly, low cost building material and another one is for Best woman engineering college teacher for the year 2012. She has produced 11 doctorates and guiding 7 Ph.D scholars. Recently she has been honoured with prestigious Research Award by UGC, New Delhi (2014-2016). Presented two papers in International Conference at Hong Kong in October 2015. Presently she is the Professor & Dean (Research & Development) in Sona College of Technology (Autonomous) Salem, Tamilnadu, India and also established and heading the research centre SONA COIN (COncrete INnovation).