

# Effect of Incorporating Silica Fume in Fly Ash Geopolymers

Suresh Thokchom<sup>1</sup>, Debabrata Dutta<sup>2</sup>, Somnath Ghosh<sup>3</sup>

**Abstract**—This paper presents results of an experimental study performed to investigate effect of incorporating silica fume on physico-mechanical properties and durability of resulting fly ash geopolymers. Geopolymer specimens were prepared by activating fly ash incorporated with additional silica fume in the range of 2.5% to 5%, with a mixture of sodium hydroxide and sodium silicate solution having Na<sub>2</sub>O content of 8%. For studying durability, 10% magnesium sulphate solution was used to immerse the specimens up to a period of 15 weeks during which visual observation, weight changes and strength changes were monitored regularly. Addition of silica fume lowers performance of geopolymer pastes. However, in mortars, addition of silica fume significantly enhanced physico-mechanical properties and durability.

**Keywords**—Fly ash; Silica fume; Geopolymer; Apparent porosity; Sorptivity; compressive strength; durability

## I. INTRODUCTION

GEOPOLYMERS are a class of new binder manufactured by activating an aluminosilicate source material such as fly ash, silica fume, blast furnace slag etc, with a highly alkaline solution and moderate thermal curing. In the recent years, interest in geopolymer is increasing manifold due to their reported advantages over ordinary Portland cements. Geopolymer materials are reported to possess high early strength, better durability and have almost no alkali-aggregate reaction [1]. These materials are therefore projected to be cement for the future [2]. In recent years, fly ash based geopolymers have received tremendous attention for its various reported superior properties and also for its abundant availability as wastes from thermal power plants. Low calcium fly ash based geopolymer manufactured with different activators have shown high compressive strengths and excellent performance when exposed to different acid and sulphate solutions [3]-[10]. It has also been reported to be highly resistant to elevated temperatures [11]-[13]. Geopolymer activated by a mixture of sodium hydroxide and sodium silicate solution yield higher compressive strength [Bakharev 2005a,b,c]. Moreover, the microstructure development depends on alkali content of activating solution. Silica fume has been widely used in cement concrete to improve its permeability property. Brew and Mackenzie [14] manufactured geopolymer from silica fume with addition of sodium aluminate solution and reported to develop good strength. Past investigations have not used silica fume as an

additive in manufacturing geopolymers. Incorporation of silica fume into fly ash can be expected to improve the properties of resulting geopolymers in terms of strength and porosity.

The objective of the experimental investigation was to study the effect of addition of silica fume on physico-mechanical properties and durability of fly ash based geopolymer composites. Up to 5% silica fume by weight was added in increments to the fly ash while manufacturing geopolymer paste and mortars. Durability of geopolymer materials was assessed by regular monitoring of its physical appearance, weight changes and compressive strength changes on exposure to 10% magnesium sulphate solution. In addition, effects of apparent porosity and water sorptivity on weight and strength changes are studied.

## II. EXPERIMENTAL

### A. Materials

Low calcium Class F fly ash used in the present research work was collected from Kolaghat Thermal Power Plant near Kolkata, India. It had chemical composition as given in Table-1. About 75% of particles were finer than 45 micron and Blaine's specific surface was 380m<sup>2</sup>/kg. Silica fume was obtained from Oriental Trexim Pvt Ltd, Mumbai, India. The chemical composition of silica fume is given in Table 1. It has a specific gravity of 2.36 and BET surface area of 18900 m<sup>2</sup>/kg. Laboratory grade sodium hydroxide in pellet form (98 percent purity) and sodium silicate solution (Na<sub>2</sub>O= 8%, SiO<sub>2</sub> =26.5% and 65.5% water) with silicate modulus ~ 3.3 and a bulk density of 1410 kg/m<sup>3</sup> was supplied by Loba Chemie Ltd, India. The alkaline activating solution was prepared by dissolving required quantity of sodium hydroxide pellets directly into predetermined quantity of sodium silicate solution. It had Na<sub>2</sub>O content and SiO<sub>2</sub> content as 8.0% of fly ash, thereby making SiO<sub>2</sub>/Na<sub>2</sub>O ratio of 1. Water to fly ash ratio was maintained at 0.33. The activator solution was left at room temperature overnight before being used to manufacture geopolymer specimens. For preparation of exposure solution, Magnesium sulphate (MgSO<sub>4</sub>) in powder form with 99% purity was directly dissolved in water to get 10% magnesium sulfate solution (by weight).

Suresh Thokchom is with the Civil Engineering Department, Manipur Institute of Technology, Imphal 795004 India; fax+91 33-2335-7254; e-mail: thok\_s@rediffmail.com

Debabrata Dutta is the Jadavpur University, Kolkata 700032 India (e-mail: debabrata\_12@rediffmail.com).

Somnath Ghosh is with the Civil Engineering Department, Jadavpur University, Kolkata 700032 India (e-mail: som\_ghosh2000@yahoo.co.in).

TABLE I  
COMPOSITION OF FLY ASH AND SILICA FUME (% MASS)

Chemical composition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	LOI
Fly ash	56.01	29.8	3.58	1.75	2.36	0.30	0.73	0.61	Nil	0.44	0.40
Silica fume	92.00	0.46	1.60	Nil	0.29	0.28	0.61	0.51	0.19	Nil	1.00

LOI- loss on ignition

### B. Preparation of specimen and testing

In a Hobart mixer, fly ash, with or without silica fume was mixed with predetermined quantity of activator solution for 5 minutes. The geopolymer mix exhibited a thick sticky nature with good workability. In case of mortar specimens, sand in surface saturated condition was gradually introduced at this stage and continued mixing for another 5 minutes. The ratio of fly ash to sand was taken as 1. The mix was then transferred into 50 x 50 x 50 mm steel cubes. Table vibration was provided for 2 minutes to expel any entrapped air. After 60 minutes, the cubes were cured in an oven for a period of 48 hours at 85°C and then allowed to cool inside the oven [15] specimens were demoulded and stored at room temperature at a dry place before testing. Some specimen data of the present study are given in the Table.II.

TABLE II  
DETAILS OF THE GEOPOLYMER TEST SPECIMENS

Sample ID	Silica fume (% by wt. of fly ash)	Type of specimen	28 days Compressive Strength (MPa)
FP	0	Paste	37
FP1	2.5	Paste	34
FP2	5.0	Paste	30
FM	0	Mortar	26
FM1	2.5	Mortar	31
FM2	5.0	Mortar	36

After 28 days from casting, the geopolymer specimens were immersed in 10% magnesium sulphate solution for 15 weeks. At regular intervals, specimens were tested for physical changes, weight changes and compressive strength changes. An Optical microscope was used for observing surface changes. For every data point, three replicate specimens were tested. In addition, unexposed specimens were subjected to water absorption, apparent porosity, water sorptivity and compressive strength tests to assess the pore characteristics. Apparent porosity and sorptivity tests were conducted as per the procedure followed by Thokchom et al.[16].

## III. RESULTS AND DISCUSSION

### A. Unexposed geopolymer specimens

#### 1. Apparent Porosity and Water absorption

Apparent porosity and water absorption of the geopolymer specimens have been determined to assess their pore characteristics. Fig. 1 and Fig.2 presents the apparent porosity and water absorption for the specimens. It is interesting to note that apparent porosity of geopolymer paste specimens increases with increasing silica fume content. In contrast, mortar specimens exhibited decreasing apparent porosity with additional silica fume. FP specimen without silica fume

showed apparent porosity of 24.3% while for FP2 specimen with 5% silica fume, the apparent porosity increased to 25.9%. FM2 specimen registered an improvement (17.2%) over that of FM specimen (22.1%). Results of water absorption test support the porosity of the geopolymer specimens. Geopolymer pastes revealed higher water absorption with addition of silica fume. However, improvements in water absorption are observed incase of geopolymer mortars with additional silica fume. Least water absorption recorded for pastes was for FP with a value of 12.23%. Mortar specimens indicated significantly lower water absorption. The minimum water absorption for mortar specimen was found as low as 3.92% for FM2 specimen with 5% addition of silica fume. It can be concluded that addition of silica fume enhances pore characteristics for mortar specimens whereas it reduces in the paste specimens.

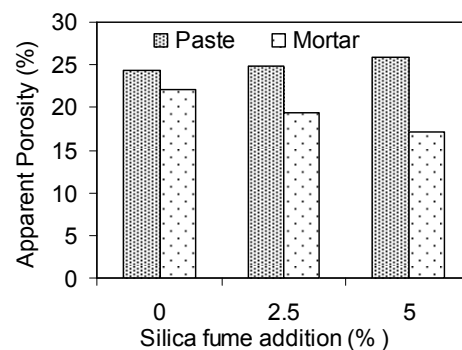


Fig. 1 Apparent porosity of Geopolymer specimens

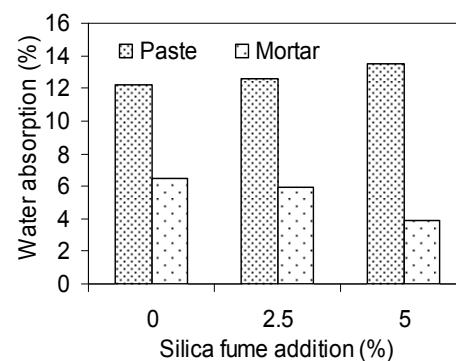


Fig. 2 Water absorption of Geopolymer specimens

2. Water sorptivity

Water ingress into a non saturated cement concrete is due to sorption, driven by the capillary forces. Sorptivity is considered to be an important property associated with durability of cement concrete specimens [17]. The test for sorptivity consists of measuring the capillary sorption of water with time. Typical curves for cumulative absorption of water plotted against square root of time are presented in Fig.3, for the geopolymer specimens. For finding sorptivity, slope for the initial linear portion for each of these curves were determined which represents the sorptivity values. Fig.4 presents sorptivity results for the geopolymer paste and mortar specimens. As observed from Fig.3, capillary sorption is initially rapid and becomes significantly low at later stages. It is evident that cumulative sorption is highest for FP2 specimen which had maximum apparent porosity. Similarly, specimen having least apparent porosity (FM2) indicates lowest cumulative sorption of water. As expected, sorptivity values for paste specimens were found to be significantly higher than those of geopolymer mortar counterparts. Addition of silica fume decreased the water sorptivity for geopolymer mortars while it caused an increase incase of paste specimens.

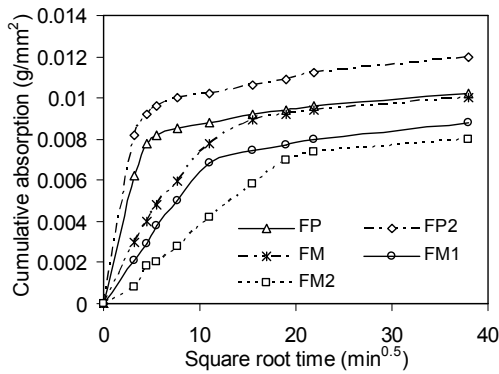


Fig. 3 Trend of cumulative sorption of water for Geopolymer specimens

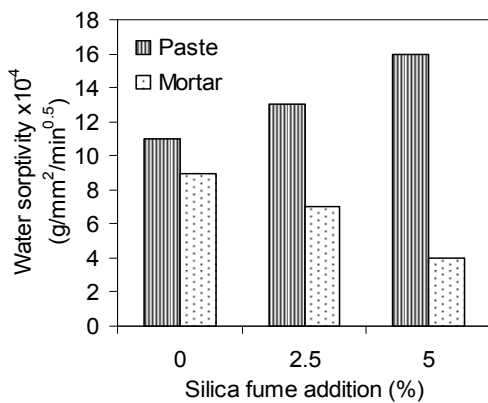


Fig. 4 Water sorptivity of Geopolymer specimens

B. Durability in Magnesium sulphate

1. Visual observation

Changes in physical appearance of geopolymer specimens after exposure to magnesium sulphate solution were monitored at pre-selected intervals. Within one week of exposure, white precipitates deposited on the surfaces which were initially soft and then became hard at later stages of exposure. Some elongated needle like crystal formations started appearing on the surfaces after few weeks. Such structures have been also reported by some authors [7],[18]-[19]. It was identified as gypsum.

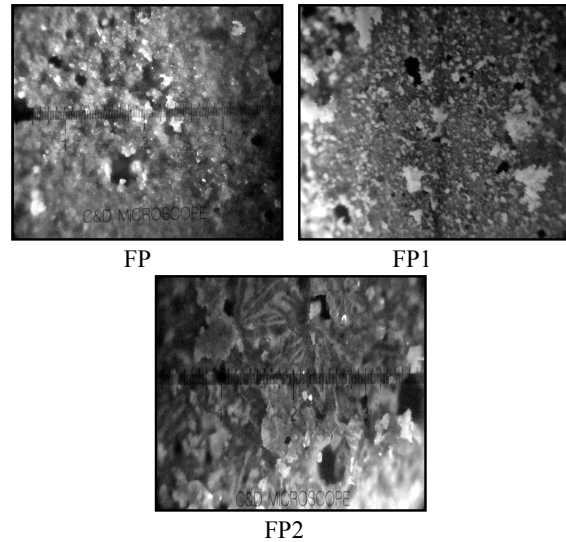
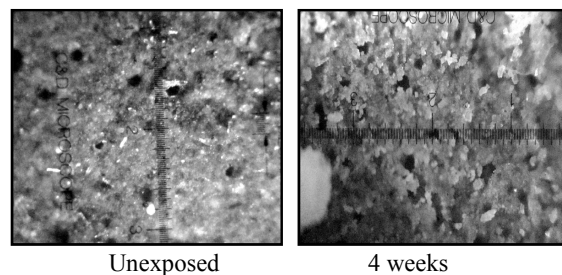
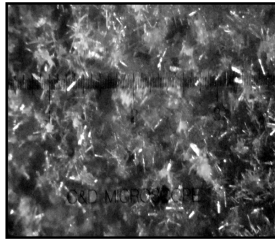


Fig. 5 Geopolymer paste specimens after 4 weeks in 10% magnesium sulphate solution

Surface deposits have been observed to increase with duration of exposure. Geopolymer specimens did not show any signs of disintegration and remained structurally intact throughout the exposure duration. Images of surfaces for paste specimens as seen under an optical microscope after 4 weeks in magnesium sulphate solution are shown in Fig. 5. These images clearly reveal the surface deposits. Elongated structures can be noticed in the image of specimen FP2. Fig. 6 presents the surface images of unexposed and exposed FM2 specimens. After 12 weeks of exposure, surface of FM2 specimen showed needle like elongated crystal formations.





12 weeks

Fig. 6 Geopolymer mortar specimen FM2 at various stages of exposure in 10% magnesium sulphate solution

## 2. Weight changes

After washing and removal of surface deposits, weights for exposed specimens were measured at regular intervals in saturated surface dry condition. The trends of weight changes for the alkali activated fly ash geopolymer specimens are presented in Fig. 7 and Fig. 8, for paste and mortars respectively. Rapid increase in weight occurred both for paste and mortar specimens up to 1 week of exposure. This is probably due to formation of reaction products such as gypsum and ettringite on the exposed surface and within the pores. Weight gain continued gradually in all the specimens till 9 weeks, except incase of FM2 specimen. It indicates continuous penetration of sulphate solution in addition to formation of reaction products due to interaction of geopolymer material with the exposure solution. However, weight of specimens began to decrease beyond 9 weeks of exposure. Incase of paste specimens, FP2 which had highest silica fume content recorded maximum drop, followed by specimens of FP1 and FP. However, among mortar specimens, maximum drop in weight was exhibited by FM specimen, followed by FM1 and FM2 specimens. The drop in weight could be due to migration of alkalis from the specimens into the solution and also due to breakdown and dissolution of some geopolymer material. It can be expected from the trend for weight change at later stages of exposure that further exposure beyond 15 weeks could lead to weight losses in the specimens. Final weight changes for the specimens were FP-8.5%; FP1-8.2%; FP2-7.3%; FM-1.7%; FM1-3.3% and FM2-4.6%. Within the present duration of exposure, maximum weight gains recorded were after 9 weeks; FP2 and FM showing maximum gains among pastes and mortars respectively. The weight changes are noticed being influenced by apparent porosity. Highest weight gains are presented by specimens with highest porosities. Incase of paste specimens, addition of silica fume into the mix exhibited poor performance in terms of weight changes. However, performance of mortar specimens incorporated with silica fume improved.

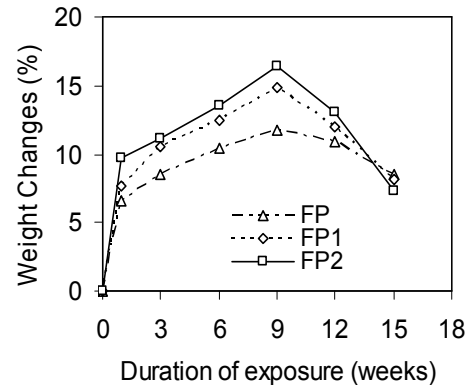


Fig. 7 Weight changes for geopolymer paste specimens

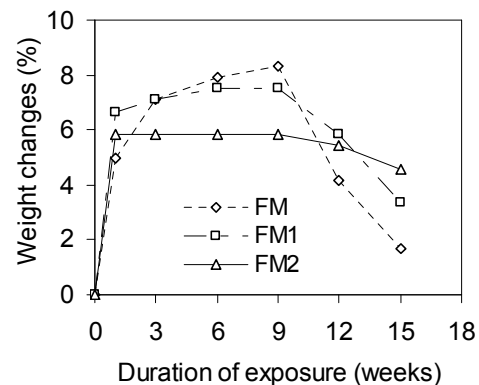


Fig. 8 Weight changes for geopolymer mortar specimens

## 3. Compressive strength changes

Fig. 9 and Fig. 10 present the variation of residual compressive strength over the entire period of exposure in 10% magnesium sulphate solution. Both paste and mortar specimens reveal similar trends. Compressive strength of sulphate exposed specimen reduced with time. However, increasing trends in strength are observed after 9 weeks of exposure. Bakharev [4] reported loss of strength due to migration of alkalis from the specimens and also due to diffusion of calcium and sulphur near the surface region. After 15 weeks in sulphate solution, paste specimens recorded residual strengths of 83.5%, 74.5% and 56.5% for FP, FP1 and FP2 specimens respectively. Residual compressive strength was higher for specimens without silica fume among the pastes. In contrast, mortar specimen incorporated with silica fume presented higher residual strengths. FM, FM1 and FM2 specimens had residual strengths of 65.4%, 87.1% and 98.5% respectively at the end of 15 weeks exposure. It may be noted that residual compressive strength of geopolymer specimens are significantly influenced by their porosities. The porosities have been found to be affected differently in paste and mortar specimens with additional silica fume.

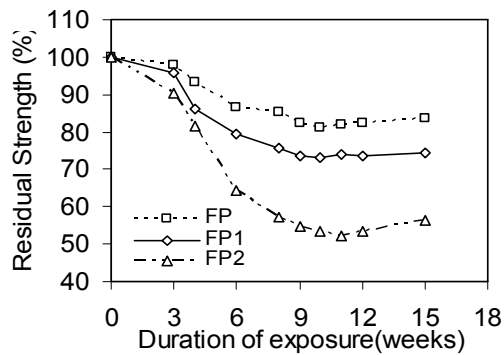


Fig. 9 Residual strength for geopolymer paste specimens

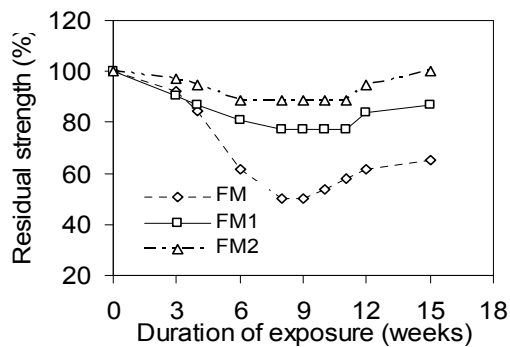


Fig. 10 Residual strength for geopolymer mortar specimens

#### IV. CONCLUSION

Based on the results of the present experimental investigation, following conclusions are drawn.

1. Addition of silica fume increases apparent porosity and water absorption of fly ash based geopolymer pastes. However, these are improved incase of geopolymer mortars.
2. Geopolymer paste incorporated with silica fume resulted in higher sorptivity while it for geopolymer mortars.
3. Compressive strength of silica fume incorporated geopolymer pastes were found lower while silica fume incorporated mortars exhibited higher strength.
4. Performance of geopolymer pastes incorporated with silica fume was poor when compared to geopolymer without silica fume. Silica fume added geopolymer pastes resulted in lower residual strength after exposure to magnesium sulphate solution.
5. Geopolymer mortars with additional silica fume showed enhanced performance in exposure to magnesium sulphate solution.
6. In comparison, geopolymer mortar specimens were found better in performance than the paste counterparts.

#### REFERENCES

- [1] J. Davitovits, "Properties of geopolymer cements," Proceedings of the first International conference on alkaline cements and concretes vol.1, SRIBM, Kiev, 1994, pp. 131-149.
- [2] A. Palomo, M.W. Gruztek, M.T. Blanco, "Alkali activated fly ashes. A cement for the future," Cement and Concrete Research 29 1999, pp. 1323-1329.
- [3] T. Bakharev, "Resistance of geopolymer materials to acid attack," Cem. & Conc. Research 35, 2005, pp. 658-670.
- [4] T. Bakharev, "Durability of geopolymer materials in sodium and magnesium sulphate solutions," Cement and Concrete Research 35, 2005, pp.1233-1246.
- [5] X.J. Song, M. Marosszeczy, M. Brungs, R. Munn, "Durability of fly ash based geopolymer concrete against sulphuric acid attack," 10 DBMC International conference on durability of building materials and components, Lyon, France, 2005.
- [6] S. Thokchom, P. Ghosh, S. Ghosh, "Resistance of fly ash based geopolymer mortars in sulphuric acid," Journal of Engineering and Applied Sciences 4(1),2009, pp. 65-70.
- [7] S. Thokchom, P. Ghosh, S. Ghosh, "Performance of Fly ash based Geopolymer mortars in sulphate solution," Journal of Engineering Science and Technology Review 3(1), 2010, pp.36-40.
- [8] S. Thokchom, P. Ghosh, S. Ghosh, "Durability of Fly ash Geopolymer mortars in Nitric acid-effect of Alkali (Na<sub>2</sub>O) content," Journal of Civil Engineering and Management 17(3),2011, pp. 393-399.
- [9] Fernandez-Jimenez, I.garcia-Lodeiro, A. Palomo, "Durability of alkali activated fly ash cementitious materials," J. Mater Sci 42, 2007, pp.3055-3065.
- [10] S.E. Wallah, B.V. Rangan, "Low calcium fly ash based geopolymer concrete: Long term properties," Research report GC2, Curtin University of Technology, Australia, 2006.
- [11] T. Bakharev, "Geopolymeric materials prepared using class F fly ash and elevated temperature curing," Cement and Concrete Research 35, 2005, pp. 1224-1232.
- [12] T. Bakharev, "Thermal behaviour of geopolymers prepared using class F fly ash and elevated temperature curing," Cement and Concrete Research 36, 2006, pp. 1134-1147.
- [13] D.L.Y. Kong, J.G. Sanjayan, "Effect of elevated Effect of elevated temperatures on geopolymer paste, mortar and concrete," Cement and Concrete Research, 40, 2010, pp. 334-339.
- [14] D.R.M. Brew, K.J.D. MacKenzie, "Geopolymer synthesis using silica fume and sodium aluminate," J Mater Sci 42, 2007, pp. 3990-3993.
- [15] R. Thakur, S. Ghosh, "Fly ash based geopolymer composites," Proceedings of 10<sup>th</sup> NCB International seminar on cement and building materials, New Delhi, India 3, 2007, pp. 442-451.
- [16] S. Thokchom, P. Ghosh, S. Ghosh, "Porosity and Sorptivity on Performance of Fly Ash Based Geopolymer Mortars in Nitric Acid", International Journal of Applied Engineering Research, 4(11), 2009, pp.2085-2092.
- [17] B.B. Sabir, S, Wild, M. O'Farrel, "A water sorptivity test for mortar and concrete," Materials and Structures, 31, 1998, pp.568-574.
- [18] A. Allahverdi, F. Skvara, "Sulfuric acid attack on hardened paste of geopolymeric cements part 1, Mechanism of corrosion at relatively high concentrations, Ceramics-Silikaty 49 (4), 2005, pp.225-229.
- [19] F. Rendell, R. Jauberthie, "The deterioration of mortar in sulphate environments. Cement & Concrete Research 13, 1999, pp. 321-327.