PERFORMANCE AND COMPATABILITY OF PERMEABILITY REDUCING AND OTHER CHEMICAL ADMIXTURES IN AUSTRALIAN CONCRETES

by Robert L Munn, Gary Kao, Zhen-Tian Chang

Synopsis: A substantial research program has been undertaken at the Australian Centre for Construction Innovation of the University of New South Wales to determine the benefits resulting from the use of permeability reducing admixtures as integral components of concrete required to demonstrate superior durability in aggressive environments. This program used commercial concretes which contained conventional water reducing admixture, different types of supplementary cementitious materials and permeability reducing admixtures at various dose rates.

The program included testing of both plastic state and hardened state properties of these concretes to assess the compatibility and impact on performance properties. Both concrete and mortar testing have been undertaken in order to determine a range of properties including setting time, strength, drying shrinkage, chloride resistance, and sulphate resistance. Assessment of these tests indicates that these permeability reducing admixtures can positively influence the key performance properties indicative of improved durability.

Keywords: durability, compatibility, permeability reducing admixture, supplementary cementitious materials

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INTRODUCTION

Durability is one of the major design criteria for concrete structures exposed to aggressive environments including water pressure, chloride and sulphate attacks. In appropriate selections of materials or mixture proportions can allow corrosion of reinforcement and eventually lead to reduction of structural capacity and service life. Durability factors important for underground and off shore structures may include 1) early strength gain; 2) drying shrinkage; 3) sulphate resistance; and 4) chloride resistance. Concrete is normally considered to be porous due to existence of capillary pores, gel pores and potentially porous cement-aggregate interface zones. Important traditional means to improve concrete durability are through reduction of water to cement ratio and/or increase of the moist curing period. Recently, many new materials and techniques have been developed to control corrosion by inclusion of inhibitors (1) or by reduction of penetration of water, chloride, and sulphate ions into concrete, eg. membrane coatings or concretes with high proportions of supplementary cementitious materials. For vary aggressive environments, such as tidal-zone and walls of underground tunnels, serial protection to restrict corrosive ion diffusion and leaking need to be taken into consideration, to minimise maintenance and repair cost. Partial replacement of Portland cement with supplementary cementitious materials (SCM) has been used widely in aggressive environments applications and the effects have been discussed and published widely in the literature (2,3).

Various types of permeability reducing admixtures are available in the market (refer to AS1478). Some of them are classified as hydrophobic admixtures due presence of long chain, fatty acids and vegetable oils (4), whereas others are classified as microstructure modifier which reduces concrete permeability through crystallisation in concrete pores (5). A substantial research program is currently undertaken at the Australian Centre for Construction Innovation (ACCI) of the University of New South Wales to investigate a broad range of properties of various commercial concrete mixes modified with the latter type of permeability reducing admixture. The major aim of this investigation is to evaluate and explore compatibility in a range of typical concretes and the optimal efficiency of these permeability reducing admixtures in enhancing concrete durability performance through improvement of the concrete microstructure.

MIXTURE PROPORTIONS AND TEST PROGRAM

Materials and Sample Preparation

To minimise the difference in performance between “lab concrete” and “site concrete” commercial concrete batches of two cubic metres each were used in this research for investigation of real concrete batches for construction applications. Three types of cement used in the concrete mixtures were AS3972 Type-SL Portland cement only, or AS3972 Type-GB fly ash blend with 20% fine fly ash or AS3972 Type-GB slag blend with 38% slag. For each type of cement, a control batch without permeability reducing admixtures was produced and at least one batch of concrete was produced with the addition of a permeability reducing admixture at various dose rates recommended by the manufacturer.

All concrete batches were supplied by a premixed concrete plant based on 32 MPa strength grade commercial concretes. AS1478 Type-WR admixture was added at a rate of 300ml/100kg cement to achieve a target slump of 80mm. Two types of permeability reducing admixture (PRA-1 and PRA-2) were added into selected concrete batches at the premixed plant according to manufacturer’s specification and recommendations. The crystal growth types of permeability reducing admixtures react with by-products of cement hydration and generate insoluble crystals within the pores and capillary (5).

The details of mixture proportions are given in Table 1, Type-SL portland cement had a Blaine fineness of 340 m^2/Kg. Type-GB cement (with 38% slag blend) had a Blaine fineness of 400 m^2/Kg. Blaine fineness of Type-GB cement (with 20% fly ash blend) was not available.

TESTING PROGRAM

This paper summarise the results of testing the concrete mixtures according to the following testing program.

Slump and Setting Time: Slump and setting time of each concrete mixture was tested in accordance with the Australian Standard AS1202.

Compressive Strength: Compressive strengths were tested with 200mmx100mm cylinder samples after curing for 3, 7, and 28 days and were tested in accordance with AS1012.9.

Drying Shrinkage: Drying shrinkage samples were cast and cured according to AS1012.13. Shrinkage was measured every 7 days until 56 day drying.

Length Change in Sulphate Solution: Sulphate resistance samples were cast and cured according to AS2350.14. Expansions of samples were measured using a comparator every 2 weeks after immersion in sulphate solution. Final readings were taken after 16 weeks of immersion.

Rapid electrical chloride ion penetration: Samples were prepared according to ASTM C1202. Test apparatus was set up in accordance with CSIRO modified ASTM 1202 method. The tests were carried out with samples of concrete mixes with Type-SL cement and those containing Type-GB cement with 20% fly ash.

ACCI Cyclic Ponding Test: The ACCI wetting-and-drying cyclic chloride penetration test is described in the Concrete Institute of Australia recommended practice on "Performance Criteria for Concrete in Marine Environments" (6). Prism samples (100x100x200mm) were cast and cured according to AS1012. This method essentially involves accelerated testing of chloride penetration into concrete under cyclic exposure conditions (12 hours drying under heating lamps and 12 hours immersion in 15% NaCl solution). The chloride penetration depth in concrete is indicated with a silver nitrate solution sprayed on the freshly fractured sample cross section after 28 cycles (28 days).

Nordtest: The Nordtest (NT BUILD 443, 1995) is an accelerated test method for assessment of accelerated chloride penetration into hardened concrete. It is based on immersion of cylinder samples in 16.5% NaCl solution for 35 days. The cylinder samples are coated with epoxy or polyurethane on all surfaces except for the top end surface. After the immersion period, powder samples are extracted at different depths from the exposed top surface for chloride content analysis and the chloride content results are used for chloride diffusion coefficient analysis. In this program, the silver nitrate solution used in the ACCI cyclic chloride penetration test is also sprayed on the split concrete samples after the Nordtest to determine chloride penetration depth.

EXPERIMENTAL RESULTS AND DISCUSSION

Setting Time

The influence of permeability reducing admixtures on initial and final setting time of concretes is summarised in Table 2 and Fig. 1. An addition of 0.8% PRA-1 or 0.8% PRA-2 has increased initial setting time of Type-SL cement concretes by 18~30% (Mixture-B and Mixture-D) whereas, at dosage of 1.2% of PRA-1 (Mixture-C), by approximately 60% compared with Mixture-A. One concrete mixture using PRA-2 (Mixture-D) also demonstrated a longer initial setting time compared to PRA-1 mixture (Mixture-B).

For the fly ash concretes, an addition of 0.8% PRA-1 in Mixture G-1 increased the initial setting time by 48% whereas for Mixture-G-2, the increase was 80% compared with Mixture-F. Concrete Mixture-E (with 1.2% PRA-2) also had the initial setting time increased by 50% compared with control Mixture-F. For the slag concretes, Mixture-J had the initial setting time increased by 40% compared with Mixture-I.

An addition of 0.8% PRA-1 and PRA-2 increased final setting time of the concrete by 14-18% (Mixture-B and Mixture-D) whereas at dosage rate of 1.2% PRA-1 (Mixture-C) it increased by 50%. Concrete using PRA-2 (Mixture-D) also demonstrated a longer final setting time than PRA-1 concrete (Mixture-B). For the fly ash concretes, addition of 0.8% PRA-1 increased the final setting time by 36% (Mixture-G-1) whereas for Mixture-G-2, it increased by 60% compared to concrete Mixture-I. Concrete Mixture-E, with addition of PRA-2 had an increased final setting time by 50% compared with control Mixture-F. Mixture-J had an increased final setting time of 30%.

Generally the results indicate that the typical retardation of concrete setting times using permeability reducing admixtures was similar to that reported in the literature where hydrophobic admixtures were used (7). Temperature development during setting was measured in a 300mm concrete cube sample insulated with expanded polystyrene foam. Results showed that the net temperature increase and the time taken to reach peak temperature during the setting process was similar to the respective control mixtures. Therefore, the concrete temperature was not considered to have influenced the setting time. High dosage rate of PRA in concrete together with using water reducing admixture resulted increase in setting time.

Compressive Strength

The influence of permeability reducing admixture on compressive strength of concretes made with Type-SL cement is shown in Fig. 2. All concrete strengths increased with time at a similar rate as shown by tests at 3, 7, and 28 days. All concrete mixtures modified with PRA (Mixture-B, C, and D) had higher strength than control Mixture-A at the same age. At the age of 3 days, all PRA modified mixtures had compressive strengths higher than control mixture by 8% to 14%. At ages of 7 and 28 days, PRA modified concrete mixtures recorded 4% to 8% higher strength than the control mixture.

Fig. 3 compares compressive strength results of the concrete mixtures containing 20% fly ash or 38% slag in the cement. Fly ash concretes (Mixtures-F, G-1 and G-2) had similar compressive strengths at each of 3 and 7 days, while PRA modified mixtures (Mixtures-G-1 and G-2) had 6% higher strength at 28 days compared with Mixture-F. PRA-2 modified mixture (Mixture-E) had higher strength by 16% to 26% at early ages whereas shown 12% at 28 days.

PRA modified slag concrete Mixture-J gave higher strengths than control Mixture-I at all ages by 2% to 13%. The early strength gains in PRA modified concretes contrast with the extended setting times. This may be explained by the presence of crystallisation in concrete pores, resulting in a finer and denser microstructure than in the control mixture.

Drying Shrinkage

Shrinkage results of concrete mixtures made with Type-SL cement are shown in Fig. 4. Concretes containing PRA-1 (Mixture-B and Mixture-C) had very similar drying shrinkage to control Mixture-A. However, lower drying shrinkage was recorded with the concretes Mixture-D which contained PRA-2. At 56 days, the drying shrinkage of Mixture-D was lower than control Mixture-A by 22%.

Drying shrinkage results of fly ash concretes (Mixtures-F, G-1, G-2 and E) are shown in Fig. 5. PRA-1 modified concretes Mixture-G-1 and Mixture-G-2 both had lower drying shrinkages than control Mixture-F by 12% to 14% at 56 days. PRA-2 modified concrete Mixture-E had similar drying shrinkages compared with control Mixture-F at each age. Fig. 6 shows the drying shrinkage results of slag concrete Mixture-I and Mixture-J. Both control Mixture-I and PRA modified Mixture-J had very similar drying shrinkages at each age tested.

Length Change in Sulphate Solution

Potential expansion of concretes in sulphate environments was assessed in accordance with AS2350.14 by immersion samples in a sulphate solution over 16 weeks. Fig. 7 presents length changes of mortar samples sieved out of Type-SL concretes and measured according to AS2350.14. Except for a similar performance between PRA-1 modified Mixture-B and control Mixture-A, all other PRA modified concretes (Mixtures-C and D) had lower expansion than the control Mixture-A at each age.

Length changes of samples of fly ash and slag concrete mixtures in sulphate solution when tested to AS2350.14 are shown in Fig. 8. Comparing two slag concrete mixtures, PRA modified concrete Mixture-J had 58% lower expansion than control Mixture-I. Each of the 20% fly ash concrete mixtures recorded excellent sulphate resistance, while PRA modified concretes Mixture-G-1 and Mixture-E had 7% and 27% less expansion respectively than control Mixture-F. Concretes with addition of permeability reducing admixtures have shown significant reductions in sulphate expansion especially when portland cement and 38% slag cement are used.

Rapid Electrical Chloride Ion Penetration (CSIRO modified ASTM-C1202)

Rapid electrical chloride ion penetration tests were undertaken according to CSIRO modified ASTM-C1202 method (8). The tests were carried out with samples of concrete mixtures with Type-SL cement and those containing Type-GB cement with 20% fly ash. Concretes Mixture-I and J using Type-GB cement with 38% slag were not assessed using this test method. The rapid electrical chloride ion penetration test results are presented in Fig. 9.

A minor reduction in coulomb values was found for Type-SL PRA modified concretes, Mixtures-B, C, and D. At dosage rate of 0.8% PRA-1 or PRA-2, Mixture-B and Mixture-D recorded an average of 10% lower coulomb values than control Mixture-A. At a higher dosage rate of 1.2%, Mixture-C with PRA-1 recorded 16% lower coulomb value than the control concrete.

Fly ash concretes were expected to have much lower electrical charges passing through the samples compared with Type-SL cement (9,10). Fly ash concrete control Mixture-F recorded 1280 coulombs which indicates significantly better resistance to chloride penetration compared with control Mixture-A. This result is confirmed in technical literature (9,10) where it is stated that concretes made with supplementary cementitious materials have reduced chloride ion permeability. PRA modified fly ash concretes (Mixture-G-1, Mixture-G-2 and Mixture-E) show further reduction in coulomb values by 26%, 41%, and 43% respectively compared with already low charge of control Mixture-F in the test.

The reduction of charge passed was suggested to result from depletion of OH^- ion in pore solution under the pozzolanic reaction between cement and fly ash (9). Permeability reducing admixtures modify the microstructure of concrete through crystallisation in small pores. It is suggested that these crystals have altered the chemistry of pore fluid and slightly decreased level of OH^- ions. This modification in microstructure has also enhanced the development of discontinuous pore structures and increased resistance to chloride ions migration.

ACCI Cyclic Chloride Penetration

The depths of chloride penetration in ACCI cyclic test after 28 days are shown in Fig. 11 for all three series of concretes. For concrete series with Type-SL cement only, chloride penetration depth in PRA modified concretes (Mixtures-C and D) was lower than that in control Mixture-A. Only Mixture-B had a slightly higher chloride penetration depth than control Mixture-A.

As shown in Fig. 10 fly ash concretes and slag concretes had much better resistance to chloride penetration than Type-SL cement concretes. PRA-1 modified fly ash concretes (Mixture-G-1 and G-2) were found to have chloride penetration depths 18% and 22% lower respectively than control Mixture-F. Similar reduction of chloride penetration depth of 18% was also observed in Mixture-E. Both slag concretes had good resistance to chloride penetration, while concrete Mixture-J containing PRA-1 recorded a 9% lower chloride penetration depth than control Mixture-I. Fig. 11 shows chloride penetration depth of Mixture-F and Mixture-E after being sprayed with silver nitrite solution.

It was difficult to obtain an approximate surface chloride concentration for calculation of the diffusion coefficient due to high deposition of sodium chloride on the surface during wetting-drying process. However, ACCI cyclic test results can be used for as indication of durability of concretes under tidal exposure. The slag concretes have shown highest resistance to chloride ions ingress. Nevertheless, inclusion of permeability reducing admixtures (PRA-1 or PRA-2) has demonstrated future improvement in the resistance to chloride penetration.

Chloride Penetration by Nordtest Method

Three of four Type-SL cement concretes (Mixtures-A, B, and C), two fly ash concretes and two slag concretes in this program were tested by Nordtest method, NT BUILD 443. Fig. 12 shows chloride penetration depths of all concretes tested. For each of three types of concrete, PRA-1 modified mixtures had lower chloride penetration depth than respective control concretes.

For Type-SL cement concrete, chloride penetration depths in PRA-1 modified (Mixture-B and Mixture-C) were 10% and 32% lower than that in control Mixture-A. The chloride penetration depth in PRA-1 modified fly ash concrete Mixture-G-1 was significantly lower (by 38%) than that in control Mixture-F. The PRA-1 modified slag concrete Mixture-J had a slightly lower (by 6%) chloride penetration than control Mixture-I.

At the end of the Nordtest, powder samples are extracted at different depths from the exposed top surface for chloride content analysis and the chloride content results are used to determine the chloride diffusion coefficient. Fig 13. (a), (b), and (c) show chloride penetration profiles. Chloride ion contents decrease more rapidly with distance from exposed face than control concrete mixtures-A, F, and I, and especially in fly ash and slag mixtures. Fig 14(d) shows calculated chloride ion diffusion coefficients using Fick's law. The chloride ion diffusion coefficients show similar trends to chloride profiles. Mixture-B and Mixture-C have a calculated 30% reduction in chloride ion diffusion coefficients whereas Mixture-G-1 shows 50% and Mixture-J, 77% over respective control concretes.

Overall, concretes with addition of permeability reducing admixtures demonstrated a reduced rate of chloride ion ingress. The reduction of chloride ion penetration is likely due to crystallisation from the chemical reactions between permeability reducing admixtures and by-products of cement hydration, which lower calcium hydroxide content and increases discontinuity of pore structure. It can be concluded that concretes with addition of permeability reducing admixtures have shown significant improvements in chloride resistance.

CONCLUSION

This research program investigates the durability and compatibility of concretes modified with permeability reducing admixtures (PRA). Two types of PRA (PRA-1 and PRA-2) with 2 dosage rates (0.8% and 1.2%) were used with three types of cement in commercial concretes with nominal strength of 32MPa. The test results and conclusions are summarised with respect to cement type as follow. Except for minor set retardation plastic state concrete properties were similar with or without permeability reducing admixtures, however the early strength gains in PRA modified concretes appear to contradict the extended setting times. :

1. For Type-SL cement concretes, mixtures modified with PRA admixtures have shown from modest to very significant improvements in hardened state properties. Early age strengths were generally improved and PRA-2 admixture exhibited significant improvement in strength at all ages. Modified concretes show equivalent or lower drying shrinkage and sulphate expansion with Mixture-D showing significantly improved performance.

   Concretes made with both PRA-1 and PRA-2 show lower charges in the CSIRO modified ASTM-C1202 test, lower penetration of chloride ions in ACCI cyclic ponding test and lower penetration and chloride diffusion rates in the Nordtest.

2. For Type-GB cement concretes (using fly ash), mixtures modified with PRA-1 and PRA-2 admixtures have shown small to significant improvements in hardened state properties. Later age strength has increased and drying shrinkage has been reduced with introduction of the PRA-1 admixture. Sulphate expansion is similar to the already low control concrete expansion. Concrete with PRA-2 show significant lower sulphate expansion compared with control mixture. Concretes made with PRA-1 admixture show lower charges in the CSIRO modified ASTM C1202 test, lower penetration of chloride ions in ACCI cyclic ponding test and lower penetration and chloride diffusion rates in the Nordtest.

3. For Type-GB cement concrete (using slag), mixtures modified with PRA-1 admixture have shown significant improvements in hardened state properties. Strengths at all ages were modestly improved whilst drying shrinkage was similar to the control concrete. Sulphate expansion was significantly reduced and chloride penetration in the ACCI cyclic ponding test reduced with the inclusion of PRA-1 admixture in slag concretes.

Test results confirm that PRA-1 and PRA-2 admixtures are compatible with both portland and blended cement concretes which also contain a typical water reducing admixture. Overall, permeability reducing admixtures (PRA-1 or PRA-2) have contributed to significant improvements in durability of concretes.

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Table 1. Details of Concrete Mixture Proportions

Mixture Code	W/C Ratio	Cement Type and Content (kg)	Permeability Reducing Admixture (% of Cement Content)	Total Fine/Aggregate	Total Coarse/Aggregate
A	0.55	GP (330)	Nil	42%	58%
B	0.55	GP (330)	0.8% PRA-1	42%	58%
C	0.55	GP (330)	1.2% PRA-1	42%	58%
D	0.55	GP (330)	0.8% PRA-2	42%	58%
F	0.50	20% Fly Ash (360)	Nil	41%	59%
G-1	0.50	20% Fly Ash (360)	0.8% PRA-1	41%	59%
G-2	0.50	20% Fly Ash (360)	1.2% PRA-1	41%	59%
E	0.50	20% Fly Ash (360)	1.2% PRA-2	41%	59%
I	0.55	35% Slag (330)	Nil	42%	58%
J	0.55	35% Slag (330)	0.8% PRA-1	42%	58%

Table 2. Casting Conditions and Setting Time

Mixture Code	Ambient Temperature (°C).	Relative Humidity (%)	Concrete Temperature (°C).	Setting Time (hr : min)	
				Initial	Final
A	16.3	75.5	20.0	5:00	7:10
B	13.6	46.1	17.0	5:55	8:10
C	16.1	60.6	17.0	8:00	11:00
D	15.5	37.3	18.2	6:30	8:30
F	17.0	55.4	19.0	5:10	6:50
G-1	24.1	35.8	26.0	7:40	9:20
G-2	24.1	35.8	26.0	9:25	11:00
E	17.5	64.1	22.0	7:50	10:30
I	14.5	66.5	18.0	5:20	8:10
J	14.5	52.5	17.5	7:40	10:50

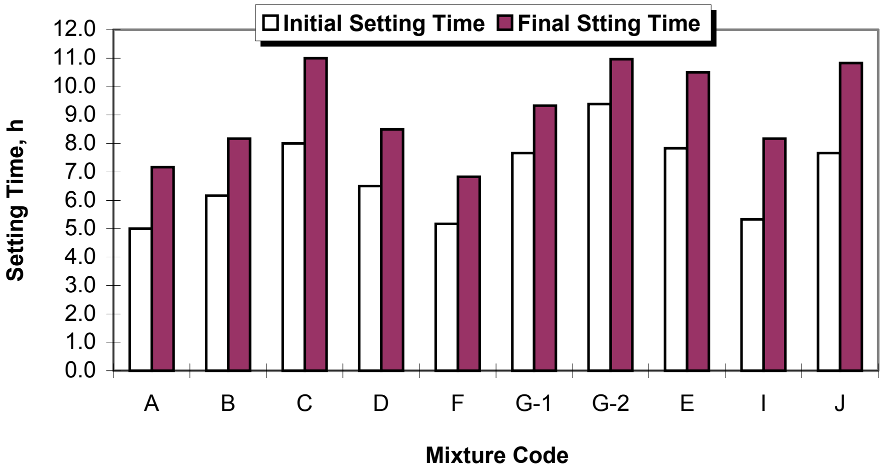


Fig. 1. Summary of Setting Time Results

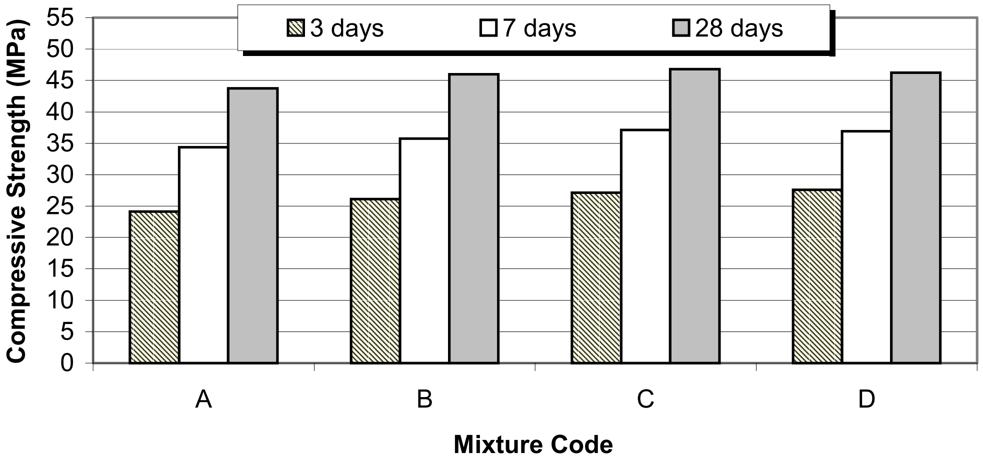


Fig. 2. Compressive Strengths of Type-SL Concretes

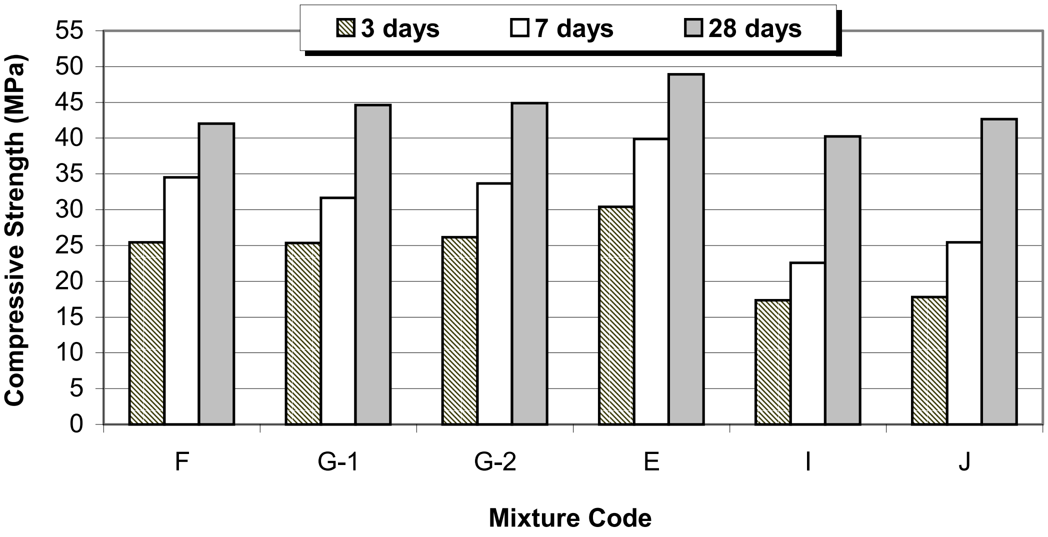


Fig. 3. Compressive Strengths of Fly Ash and Slag Concretes

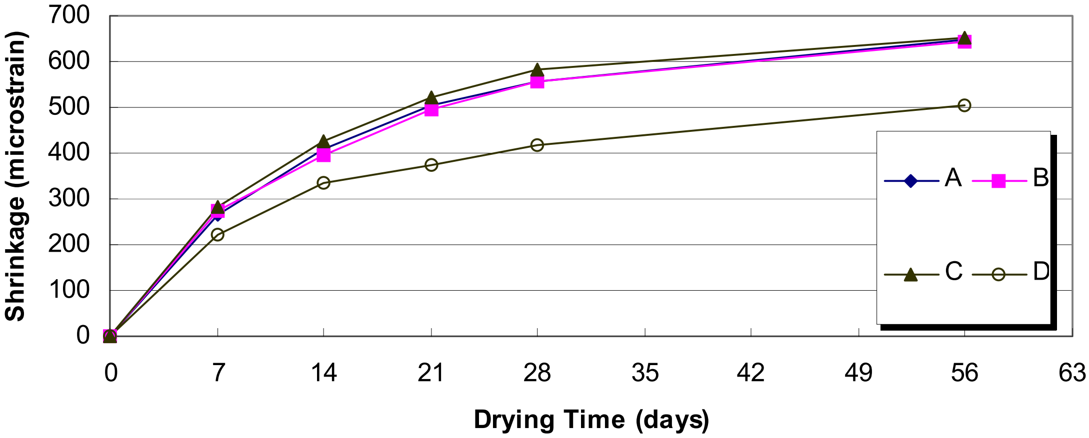


Fig. 4. Shrinkage Results of Type-SL Concretes

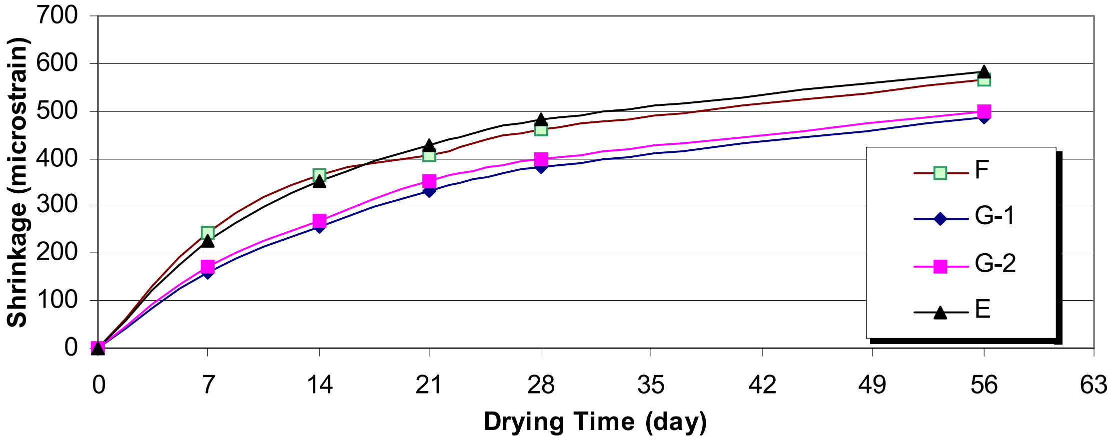


Fig. 5. Shrinkage Results of Fly Ash Concrete Mixtures

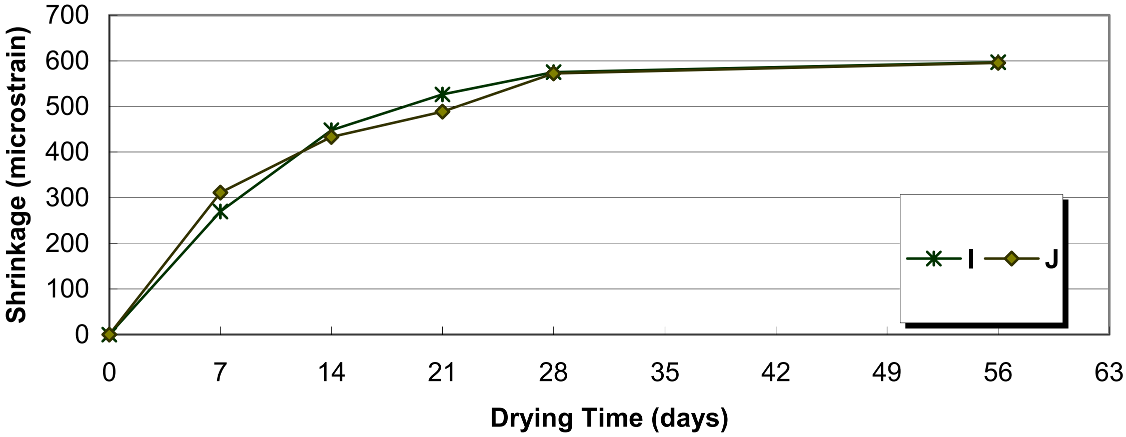


Fig. 6. Shrinkage Results of Slag Concrete Mixtures

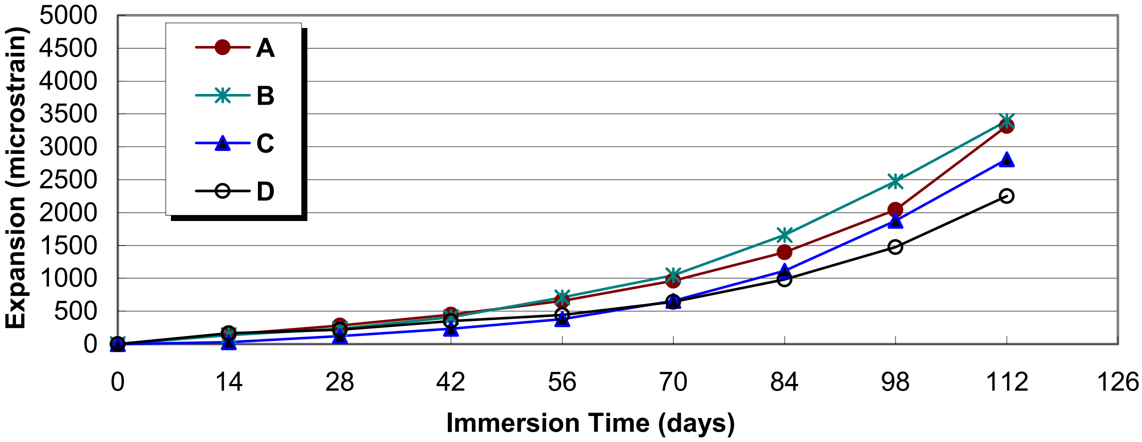


Fig. 7. Length Changes in Sulphate solutions for Type-SL Mixtures

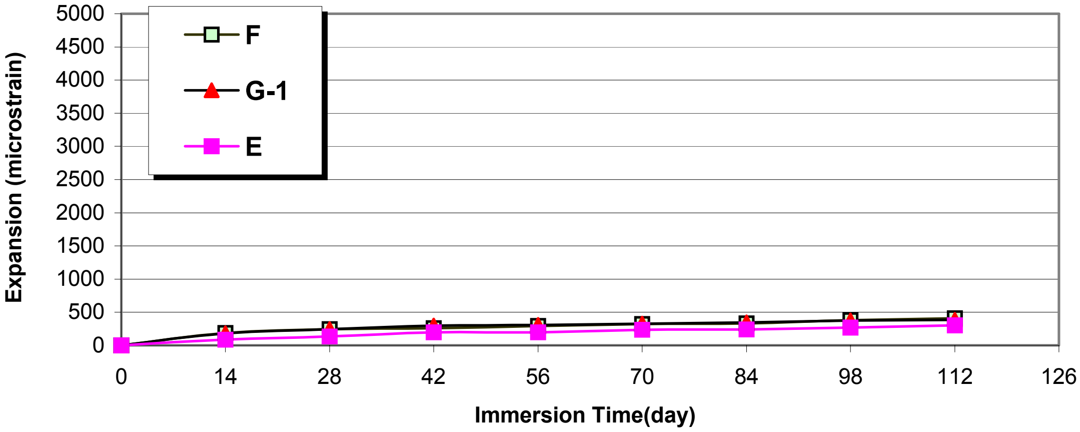


Fig. 8. Length Changes in Sulphate Solution for Fly Ash and Slag Mixtures

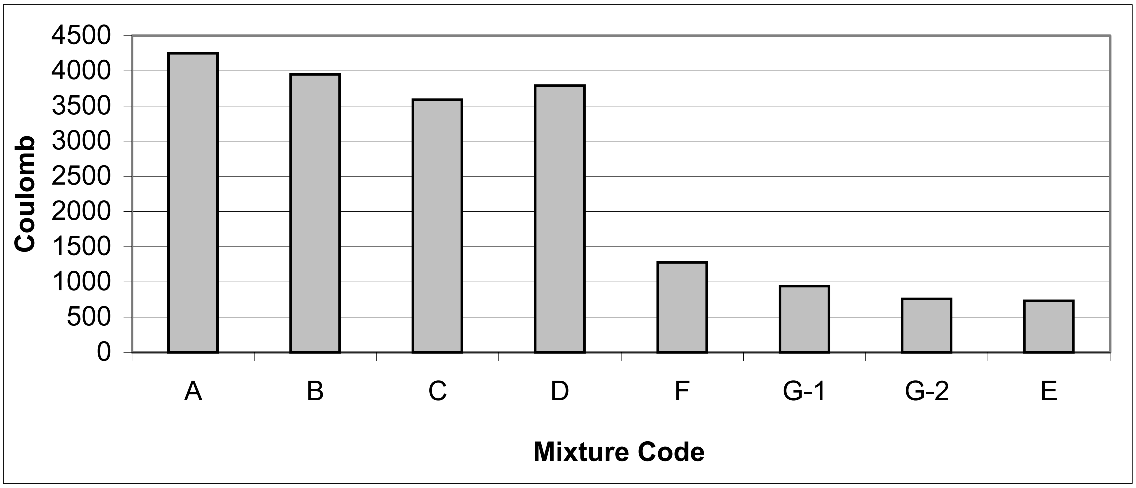


Fig. 9. Results of CSIRO Modified ASTM C1202 Testing

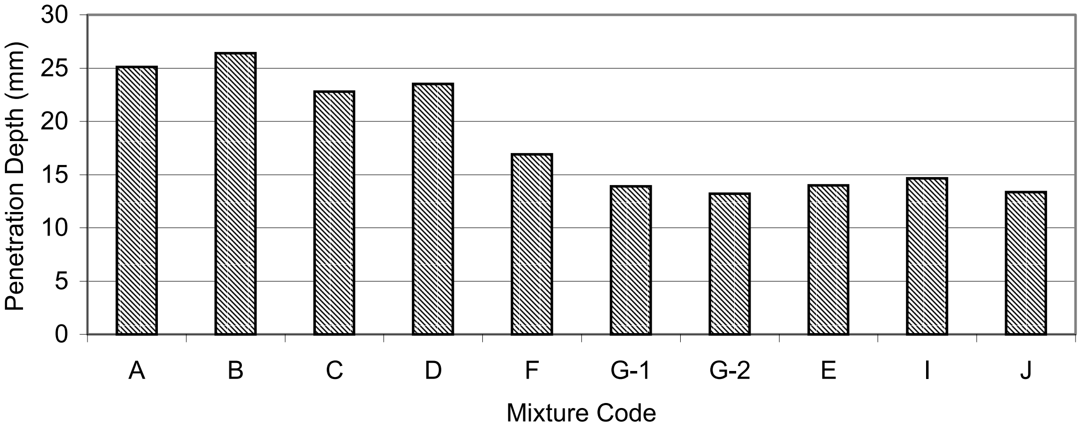
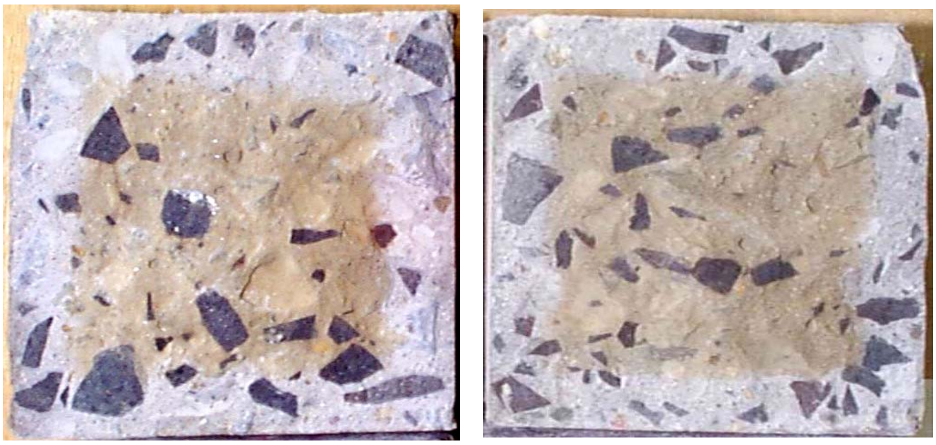


Fig. 10. Chloride Penetration Depths in Concrete after 28 days Cyclic Testing



Mixture-F **Mixture-E**

Fig. 11. Chloride Penetration Depths in Concrete

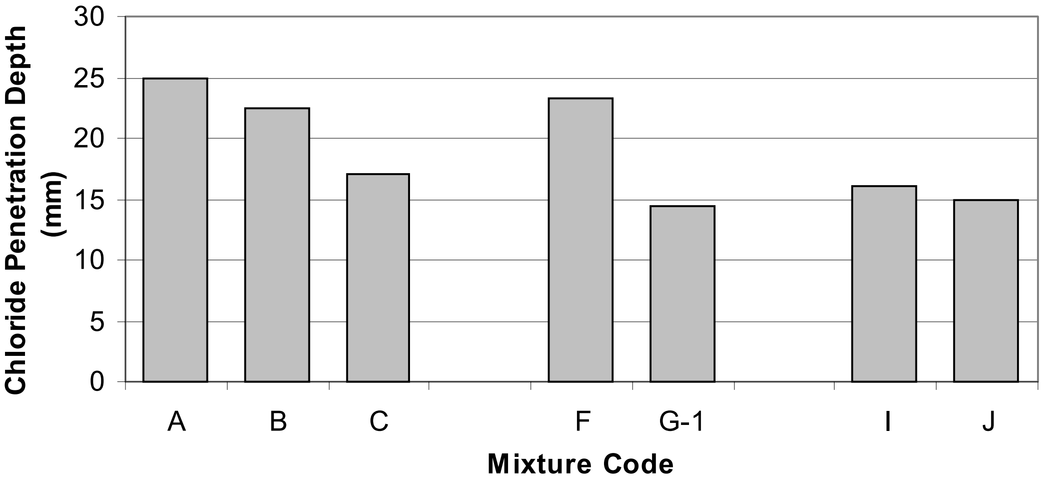
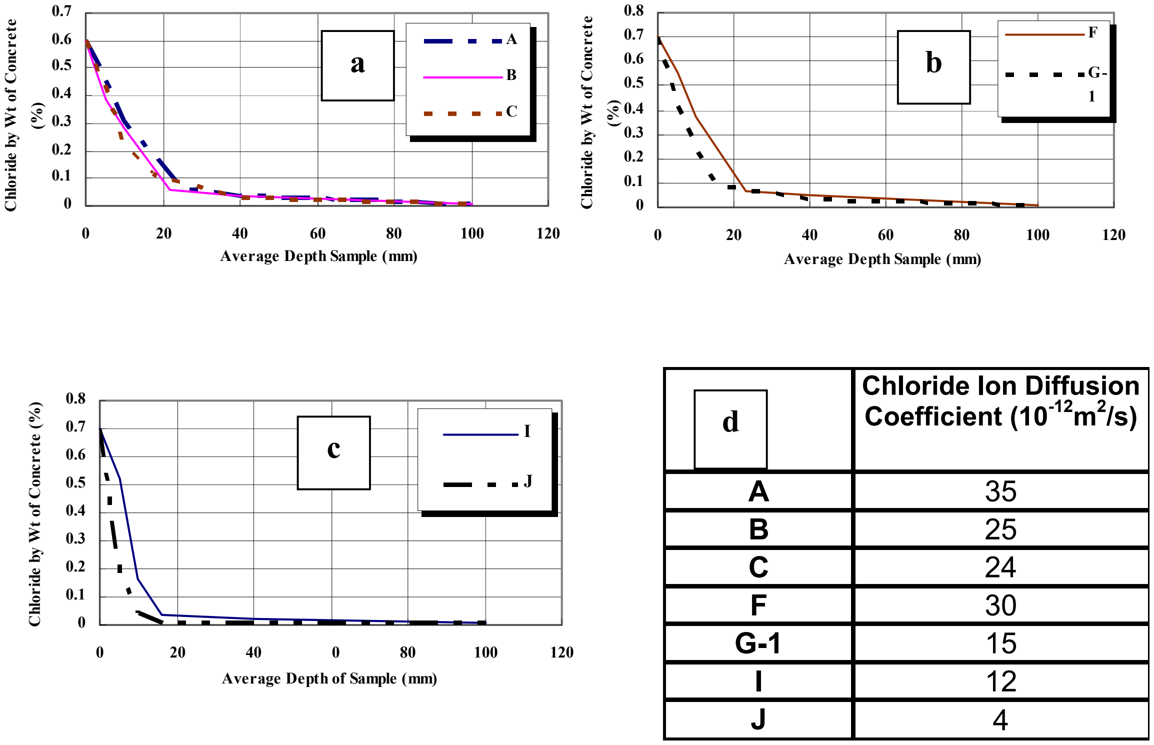


Fig. 12. Chloride Penetration Depths by Nordtest after 35 Days Immersion



d	Chloride Ion Diffusion Coefficient ($10^{-12}m^2/s$)
A	35
B	25
C	24
F	30
G-1	15
I	12
J	4

Fig 13. a), b) & c) Chloride Penetration Profiles by Nordtest; and d) Chloride Ion Diffusion Coefficients for all Mixes