Influence of Internal Curing Materials on Durability Properties of High-Performance Concrete

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***Abstract*—Proper curing of concrete is an essential requirement for achieving the desired long-term properties of concrete. Curing of concrete using Internal Curing Materials (ICMs) is considered as an effective method to eliminate self-desiccation and autogenous shrinkage. Two different types of internal curing materials (ICMs), pre-saturated recycled aggregates of construction and demolition wastes (RA), and Superabsorbent Polymers (SAPs) were used in this investigation to study the impact of these materials on the durability performance of concrete. Durability was assessed by measuring the resistance of concrete to chloride ion penetration, and chloride diffusion of high performance concrete (HPC) mixes prepared with a water to cement ratio 0.35, after 28 days of curing in both water and air. Overall results showed that the concrete specimens with air curing method have less resistance to chloride penetration compared to water cured specimens. Also, the mix with recycled aggregates under both curing methods had shown better resistance to chloride ion penetration than the control mix and mix with SAP due to the internal water effectively provided by the recycled aggregates during hydration.**

***Index Terms*—acid soluble chloride content, chloride diffusivity coefficient, chloride permeability, recycled aggregates, superabsorbent polymer, high performance concrete**

I. INTRODUCTION

In addition to workability and strength, durability is considered one of the major properties for ensuring the serviceability requirements of concrete structures. Nowadays, the research studies are mainly focused on the effect of penetration of water, chloride, and other aggressive ions into concrete or cracked concrete. As the penetration of these aggressive materials can cause serious deterioration of concrete related to the corrosion of steel in reinforced concrete structures, research has been conducted to improve the durability and long-term performance of concrete structures [1]. One approach to improve the durability of a concrete structure is to use High Performance Concrete (HPC) mixes with supplementary cementitious materials (SCMs) and low water/cement binder mass ratio (w/b), which can clearly decrease the maintenance costs and improve the service life [2]. So, HPC is currently used in a number of applications such as marine construction, high-rise buildings, bridge decks and piers, thin- wall shells, airport pavements, and others [2]. Although the HPC lead to an increase in strength and durability, the use of a low water/ cement ratio can cause early-age cracking due to autogenous shrinkage [2].

To reduce the autogenous shrinkage of HPC and to prevent its early age cracking, internal curing material (ICM) are used in the HPC mix. Many research studies proved that ICMs are capable to eliminate or considerably diminish autogenous shrinkage [2, 3]. Moreover, the lack of proper external curing could adversely affect the quality and performance of concrete structures during extreme climates. So, Internal curing or self-curing technique can be used to provide additional moisture in concrete for more effective hydration of cement and reduced self-desiccation with judicious use of available water [4,5].

The internal curing materials used for this study are pre-saturated recycled coarse aggregate (RA) and superabsorbent polymer (SAP) to supply adequate internal water that can counteract the effect of self-desiccation. The main factors which could impact the overall performance of concrete under internal curing are w/c ratio, maturity, and amount of SAP and RA [6]. The effects of using different ICMs such as SAP and pre-saturated RA on improving the long–term performance of concrete are reported in this paper. Thus, the durability properties are investigated through micro-structure related properties of concrete, such as, permeability and chloride diffusion.

II. EXPERIMENTAL DETAILS

A. Materials

The materials used for this study to produce high performance concrete were ordinary Portland cement (OPC), washed sand, normal coarse aggregates (NCA), and superplasticizer (SP), which were purchased from the

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local market. The internal curing materials that were chosen for this study were superabsorbent polymer (SAP),which was imported from outside the country, and recycled coarse aggregates (RA) from the local market. The amount of SAP was selected based on its absorption capacity as determined using the Tea Bag test method as suggested by RILEM TC 260 RSC [7]. The bulk density of SAP is 0.7 g/ml, and the specific gravity is 2.7-3.1. The quantity of SAP to be added was determined based on the absorption capacity of SAP. The water absorption of NCA and RA of 10 mm were 0.77% and 6.7 %, respectively.

B. Mixing, Demoulding and Curing

Proper mixing procedures and adequate curing are the most essential requirements for achieving high-performance concrete. Mix proportioning with ICMs is based on the principle that the volume of chemical shrinkage is compensated with the internal curing water. In this study, a high-performance concrete mix was designed for a strength of 50 MPa. The influence of different types of ICMs on the concrete properties was evaluated by preparing the mix design for the control mix and test mixes. Accordingly, the mix proportioning with ICMs was carried out as suggested by earlier researchers [8].

Three concrete mixes with a w/c ratio of 0.35 were developed for the study. This included the control mix, the mixes with two different types of ICMs; SAP, and RA. Accordingly, the mixes were designated as follows: Mix 1- C35 - Control mix with 0.35 w/c ratio; Mix 2 - C35SAP - mix with SAP at 0.35 w/c; Mix 3 - C35RA - mix with RA at 0.35 w/c.

A two-stage mixing procedure was adopted during the preparation of mixes with RA [9]. Two different mixing procedures were adopted in this study to understand the influence of mixing on the self-curing properties of RA, and the test results were reported earlier [10]. The mixing procedure that had better performance from the previous study was selected for preparing concrete mixes with RA. Based on this, the replacement of NCA with 30% RA was used in this study.

The concrete was mixed in a mixer of 50-liter capacity in the laboratory. The mixing time was about 5–7 min. The first stage of the mixing procedure was dry mixing of all ingredients for one minute. After adding water, mixing was done for another 2 minutes. Some quantity of SP was also added in this stage as SAP has to be added after adding all other ingredients. Mixing was done for at least 5 min after adding SAP. In the case of RA, pre-soaking was done for 24 hours to achieve maximum degree of saturation. The required number of cylindrical samples were prepared, and the demoulding was done after 24 hr. of casting. Two curing methods were used for this study, air and water curing. This was done to study the impact of ICMs on the properties of concrete mixes under two extreme curing conditions compared to the control concrete mix.

III. Test Program

A. Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration Test

The test for determining the resistance of concrete to chloride ion penetration was conducted according to ASTM C1202-2022 [11]. Three cylindrical samples were prepared for each concrete mix, with a diameter of 100 mm and a height of 200 mm. The specimens were cured at 28 days of curing age, using two different curing methods, air, and water curing. A concrete disc of 50 mm height was cut from the middle of each cylinder and was epoxy coated on the side. This disc was specially prepared to ensure that all concrete pores were saturated with water. A cell was constructed of the concrete disc with sodium hydroxide on one face and sodium chloride on the other, and connected to the 60-v power supply. The test was conducted on three samples, and the average value of charge passed was noted.

B. Chloride Diffusivity Test

In addition to the ASTM C1202 test, chloride diffusivity test was conducted to evaluate the integrity of concrete cover in resisting the diffusion of chloride ions, according to ASTM C1556-11a (2016) [12]. In this test, 8 concrete cylinders of 100 mm in diameter and 200 mm in height were cast from each mix, 4 cured in water and the other 4 cured in air for 28 days, and then the cylinders were epoxy-painted on all sides, except the finished surface, to allow one-dimensional penetration of chloride ions into the concrete. The cylinders were immersed in NaCl solution with a concentration of 165 g NaCl per liter of solution. When concrete is in a saturated state, chloride ions will penetrate into the concrete due to the concentration gradient between the exposed surface and the pore solution of the cement matrix. This process is often described by Fick's second law of diffusion [13]:

$$\frac{dC(x,t)}{dt} = D_c \frac{d^2[C(x,t)]}{dx^2} \tag{1}$$

Where $C(x,t)$ is the chloride concentration at depth x and time t, and D_c is the diffusion coefficient. The solution of this differential equation of chloride diffusion, using the error function, is from the surface [14]:

$$C(x,t) = C_s\left\{1 - erf\left[\frac{x}{\sqrt{4D_c t}}\right]\right\} \tag{2}$$

Where,

C_s = The chloride concentration on the concrete surface given as the percentage weight of cement, and erf = The error function.

In the first stage of this test, the initial chloride content of samples before immersing the samples in the test solution was measured, and after that the concrete cylinders were immersed in the NaCl solution. After completing the immersing period of 6 months, the chloride profile was calculated.

IV. Results and Discussions

A. Rapid Chloride Permeability Test

The test was performed by monitoring the amount of electrical current passed through the 50-mm thick slices for 6-h. period. The total charge passed in coulombs is related to the resistance of the specimen to chloride ion penetration. The chloride ion penetrability in concrete specimens with respect to the charge passed, is classified in Table I according to ASTM C1202. The results of ASTM C1202 test are shown in Table II and Fig. 1.

TABLE I. Chloride Ion Penetrability Based on Charge Passed

Charge Passed (Coulombs)	Chloride Ion Penetrability*
>4,000	High
2,000 – 4,000	Moderate
1,000 – 2,000	Low
100 – 1000	Very Low
<100	Negligible

*Source: ASTM C 1202-22

TABLE II. ASTM C1202 Results of Concrete Mixes for Different Curing Methods

	Curing Method	MIX 1 C35	MIX 2 C35SAP	MIX 3 C35RA
Charge Passed (Coulombs)	Air	3809	4329	3524
Permeability Class (ASTM C1202)	Air	mod.	High	mod.
Charge Passed (Coulombs)	Water	3224	3427	2926
Permeability Class (ASTM C1202)	Water	mod.	mod.	mod.

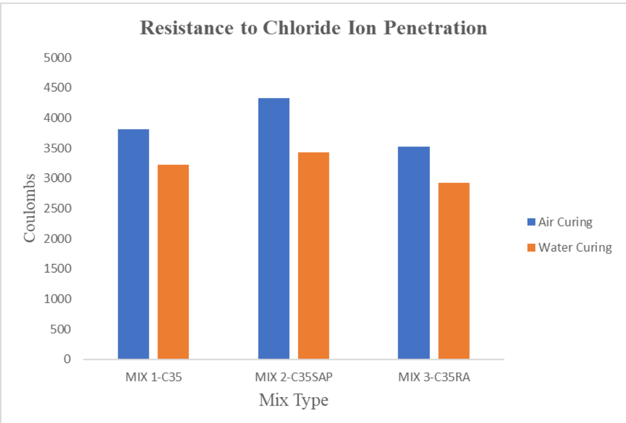

Figure 1. ASTM C1202 test results for the concrete mixes for different curing methods.

From Table II and Fig. 1 and reference to Table I (ASTM C1202), overall results showed that the concrete specimens with the air curing method have less resistance to chloride penetration, which pointed to the importance of the proper curing of concrete for the chloride permeability reduction. Also, it was clear from Table II, and Fig. 1 that the mix with SAP showed the movement of more ions indicating high charge, especially for air cured specimens, while the other mixes showed moderate coulombs. However, it was reported earlier that the addition of chemical admixtures such as SAP in a concrete mix can lead to passing additional ions, resulting in invalid results while conducting ASTM C1202 test [11]. This is mainly due to the chemical composition of the pore solution, rather than the actual permeability. So in the case of mix with SAP, the charge passed in coulombs indicates the total ionic exchange in the pore solution, and not the chloride ions' movements alone, which demonstrate the electrical conductivity of the sample.

Fig. 1 shows that both control specimens and concrete with RA have moderate chloride penetrability; however, from Fig. 1, chloride penetrability in RA concrete is less than the control mix in both the air cured and water cured specimens. Thus the mix with recycled aggregates under both curing had shown more resistance to chloride ion penetration than the control mix may be due to the improvement in the microstructure of concrete due to internal curing.

B. Determination of Acid Soluble Chloride Content

The results of initial acid soluble chloride ion content in all mixes are shown in Table III. After six months of immersing the concrete cylinders in the test solution, the samples were recalled to extract powder samples at different depths (0 to 10 mm, 10 to 25 mm, 25 to 35 mm, and 35 to 50 mm) after drying to measure the acid soluble chloride ion content according to ASTM C1152 [15].

Table IV gives the chloride limits for new construction proposed by ACI Committee 222. From Table III and Table IV, the initial chloride content in all mixes was less than the maximum allowable acid Soluble chloride ion content in concrete which is 0.2%.

TABLE III. Initial Chloride Content in Each Mix

Mix Type	Cast Date	Test Date	Initial chloride content (%)
C35-A	6/9/2021	13/10/2021	0.0045
C35-W	6/9/2021	13/10/2021	0.005
C35SAP-A	13/9/2021	13/10/2021	0.0073
C35SAP-W	13/9/2021	13/10/2021	0.0077
C35RA-A	20/9/2021	25/10/2021	0.013
C35RA-W	20/9/2021	25/10/2021	0.015

TABLE IV. Chloride Limits for New Construction Proposed By ACI Committee 222

Category of reinforced Concrete	Maximum acid soluble chloride ion content in concrete (% by weight of cement)
Pre-stressed Concrete	0.08
Non-Prestressed, Wet conditions	0.1
Non-Prestressed, Dry conditions	0.2

After immersing the concrete specimens in NaCl solution for six months, the average chloride absorbed by each mix at each depth is shown in Table V and Fig. 2.

TABLE V. AMOUNT OF CHLORIDE ABSORBED IN DIFFERENT CONCRETE MIXES AFTER IMMERSING IN NACL SOLUTION FOR SIX MONTHS

Depth	Mix Type					
(mm)	C35-A	C35-W	C35SAP-A	C35SAP-W	C35RA-A	C35RA-W
0 to 10	0.3488	0.3117	0.4327	0.5123	0.6270	0.6317
10 to 25	0.0522	0.0347	0.1234	0.1290	0.2637	0.1383
25 to 35	0.0115	0.0095	0.0277	0.0116	0.0107	0.0350
35 to 50	0.0090	0.0090	0.0164	0.0126	0.0053	0.0347

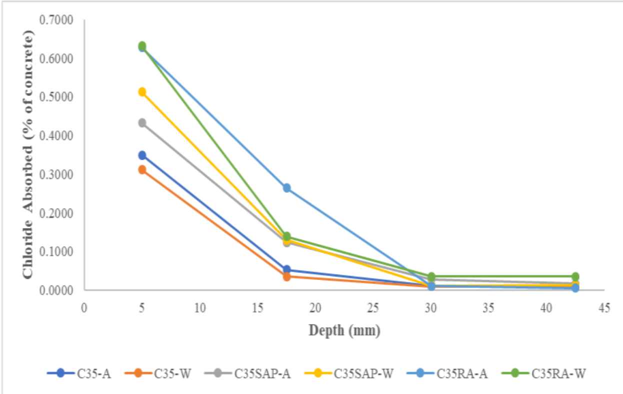

Figure 2. Chloride absorbed in each mix after immersing in NaCl solution.

From Fig. 2, the Control mix showed less chloride content at shallow depths of concrete, however, for depth of 30 mm and more, the chloride content in SAP and RA specimens were slightly more or almost the same as the control specimens. Also, from Table V, air cured RA concrete specimens showed the least chloride diffusivity among all specimens at a depth of 35-50 mm, though, at the surface, the chloride content in RA specimens was high. These results coincide with the results of ASTM C1202.

After modeling of chloride diffusivity test results using Fick's second law, the chloride diffusivity coefficient and surface chloride content for all mixes were obtained and given in Table VI. It was clear from the test results that the chloride diffusivity coefficient of different concrete mixes was comparable. However, air cured specimen for each mix showed a slightly higher chloride diffusion coefficient than the water-cured specimens. This coincides with the results of the previous test and shows the importance of properly curing concrete specimens.

TABLE VI. CHLORIDE DIFFUSIVITY COEFFICIENT AND SURFACE CHLORIDE CONTENT FOR ALL MIXES

Parameters	C35-A	C35-W	C35SAP-A	C35SAP-W	C35RA-A	C35RA-W
Chloride Diffusivity coefficient at 6 months(m^2/s)	5.941E-12	5.711E-12	7.406E-12	6.032E-12	6.308E-12	5.864E-12
Chloride Diffusivity coefficient at t=1 yr (m^2/s)	4.948E-12	4.756E-12	6.168E-12	5.023E-12	5.253E-12	4.884E-12
Surface Chloride Content (% by weight of cement)	2.8957	2.5468	3.7896	4.6403	6.3377	6.9561

V. CONCLUSION

This study investigated the influence of SAP and RA as internal curing materials on the resistance of concrete to chloride ion penetration through two different test methods. The ASTM C1202 test results showed that compared to SAP, RA had performed better in terms of resistance to chloride ion penetration. In the ASTM C1202 test, the charge passed in coulombs through SAP concrete specimens indicates the ionic exchange in the pore solution; not the chloride ions movements alone. In addition, both control specimens and concrete with RA had moderate chloride penetrability; however, chloride penetrability in RA concrete was less than the control mix in both the air cured and water cured specimens.

In the chloride diffusivity test, after 6 months of exposure, the control mix showed less chloride content at shallow depths of concrete, however, for depth of 30 mm and more, the chloride content in SAP and RA specimens were slightly more or almost the same as the control specimens. In addition, the chloride diffusivity coefficient results in three different concrete mixes were compatible. However, air cured specimens for each mix showed slightly higher chloride coefficient than the water-cured one. This coincides with the results of the ASTM C1202 test results and this indicates the importance of curing concrete specimens.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

All authors were involved in the experimental work including testing and analysis and contributed to the preparation of the paper.

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