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CHARACTERISTICS OF CHLORIDE DIFFUSION AND PORE VOLUME IN CERAMIC WASTE AGGREGATE MORTAR CONTAINING GGBS

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ABSTRACT: Ceramic waste aggregates (CWAs) were made from electric porcelain insulator wastes supplied from an electric power company, which were crushed and ground to fine aggregate sizes. The CWA mortar as an eco-efficient has been investigated containing ground granulated blast-furnace slag (GGBS). The water-to-binder ratio (W/B) of the CWA mortars was varied at 0.4, 0.5, and 0.6. The GGBS, which enhances the chloride ingress resistance, was utilized as a supplementary cementitious material. The CWA mortars partially replaced by the GGBS at 20% and 40% were immersed into a 5% NaCl solution for 48 and 96 weeks. The chloride diffusion and the pore size distribution were assessed by using an electron probe microanalysis (EPMA) and a mercury intrusion porosimetry at each immersion time. The resistance to the chloride ingress of the CWA mortar was effective in proportion to the GGBS replacement level. The changing of the apparent chloride diffusion coefficients except for the CWA without the GGBS at the W/B of 0.6 was small along the immersion time. Moreover, the apparent chloride diffusion coefficient was well related to the cumulative pore volume less than 0.1 μm of pore diameter.

Keywords: Ceramic Waste Aggregate, GGBS, Mortar, Chloride Diffusion, Pore Size Distribution

1. INTRODUCTION

Ceramic wastes discarded worldwide from ceramic industries, demolition/construction sites, electric power companies, and railway companies are one of the materials possibly recyclable as aggregates and/or pozzolans. The utilization of the ceramic wastes has been investigated by many researchers [1]-[15]. In the existing literature [2], [3], [9], [10], however, there is a shortage on the utilization of ceramic waste aggregates (CWAs) recycled from the electric porcelain insulators. They have reported that the CWAs in concrete/mortar showed no negative influence on mechanical and permeation properties. The authors have also showed on the compressive strength and the resistance to chloride ingress on the CWA mortars [16]-[18]. The CWA mortar reduces the chloride ion penetration when compared with river sand mortar. In the aggressive environment, however, the chloride resistance of the CWA mortar is still lower.

It is well-known that a mineral admixture of ground granulated blast-furnace slag (GGBS) with partial replacement is advantage in the resistance to aggressive chemical action. To enhance the chloride resistance of the CWA mortars, the authors have studied on the CWA mortars containing GGBS [19], [20]. In the previous study [19], the chloride ingress tests were performed on the CWA mortars, which have the water-to-binder ratio (W/B) of only 0.5, partially replaced with the GGBS at 15, 30, and 45% by mass. The GGBS significantly decreased the chloride ion penetration and the changing of the apparent chloride diffusion coefficients were relatively small along the immersion time up to 96 weeks.

The CWA mortars with further wide range of W/B, i.e., 0.4, 0.5, and 0.6 partially replaced with the GGBS at 20 and 40% by volume were investigated. The mechanical properties and the chloride diffusion at 48 weeks immersion in a 5% NaCl solution have been presented in [20]. In the present study, the chloride diffusion and the pore size distribution at 96 weeks immersion were reported including the results at 48 weeks immersion shown in [20].

2. EXPERIMENTAL PROGRAMS

2.1 Materials and Mixture Proportions

Electric porcelain insulators were recycled to CWAs at a recycle plant of The Kanden L&A Co., Ltd., Japan via the processes of crushing and grinding. After obtaining the blunt edge CWAs through these processes, the particle size ranging from 0.075 to 5.0 mm by sieving was used as fine aggregate. The specific gravity was 2.40. The cement was ordinary Portland cement (OPC) with the specific gravity of 3.15 and the specific surface area in Blaine of 3360 cm^2/g. The GGBS supplied from a slag cement company was used with the specific gravity of 2.91 and the specific surface area in Blaine of 6230 cm^2/g. The chemical and physical properties of these materials are given in Table 1.

The mixture proportions of CWA mortars were designed by volume as presented in Table 2. The W/B was almost 0.4, 0.5, and 0.6 and the cement was replaced by the GGBS at 20 and 40% by volume.

Table 1 Chemical and physical properties

Properties	OPC	GGBS	CWA
Chemical compositions (wt.%)			
SiO_2	20.68	33.80	70.90
Al_2O_3	5.28	15.00	21.10
Fe_2O_3	2.91	0.27	0.81
CaO	64.25	43.10	0.76
MgO	1.40	5.63	0.24
SO_3	2.10	–	–
Na_2O	0.28	0.28	1.47
K_2O	0.40	0.31	3.57
TiO_2	0.28	0.52	0.33
P_2O_5	0.25	–	–
MnO	0.09	0.20	–
SrO	0.06	–	–
S	–	0.77	–
Cl	0.015	0.004	–
Loss on ignition	1.80	0.05	–
Specific gravity	3.15	2.91	2.40
Specific surface area (cm^2/g)	3360	6.230	–

Table 2 Mixture proportions

Mixture	W/B (%)	Water (kg/m^3)	Cement (kg/m^3)	CWA (kg/m^3)	GGBS (kg/m^3)
CWA40-0	40.0	303	758	1095	0
CWA40-20	40.6	303	607	1095	140
CWA40-40	41.2	303	455	1095	280
CWA50-0	50.0	303	606	1211	0
CWA50-20	50.8	303	485	1211	112
CWA50-40	51.5	303	364	1211	224
CWA60-0	60.0	303	505	1288	0
CWA60-20	60.8	303	404	1288	94
CWA60-40	61.8	303	303	1288	187

2.2 Specimens

For all mixtures, the CWA mortars were prepared in a Hobart mixer of 5 L capacity. The mixing process started with the blending of the OPC, GGBS and CWA for 1 min and was followed the addition of water and further mixing for 3 min. Cylindrical specimens of 100 mm diameter and 200 mm height were prepared for a chloride ingress test at 48 and 96 weeks immersion (one specimen at each time) which were employed in an electron probe microanalysis (EPMA). After casting, all specimens were covered with a plastic waterproof sheet for 24 h. Subsequently, they were demouled and cured in a water tank at 20±2℃.

2.3 Test Methods

2.3.1 Chloride Ingress

At the age of 7 days, the specimens were cut down from 200 mm to 150 mm height with 50 mm top end discarded to eliminate the influence of segregation. After the specimens were allowed to dry in a laboratory condition at 20±2℃ for 24 h, they were epoxy coated leaving only one sawn surface free of coating and were kept for additional 24 h to cure the epoxy resin. Then, they were fully immersed in a 5% NaCl solution in hermetic tanks at 20±2℃ for 48 and 96 weeks. The NaCl solution was changed at each 3 months interval.

2.3.2 EPMA

After the immersion was completed, one specimen for each mixture was followed with the EPMA. The specimens were cut into 25 mm width and 60 mm length. By using the JEOL JXA-8200 instrument, the resized specimens were scanned to identify the amount of chloride ion at tiny single spot throughout its surface. The measurement conditions were an accelerating voltage of 15 kV, a beam current of 0.2 μA, a pixel size of 200 μm, a probe diameter of 150 μm, and the number of mapping points of 400 × 400 pixels. Subsequently, the chloride concentration profiles were obtained. The chloride concentration was averaged in the paste part along the same penetration depth at 0.2 mm intervals.

2.3.3 Pore Size Distribution

By using the same CWA mortars of the EPMA, the test pieces with 2.5 to 5.0 mm size were obtained from the center of cylindrical specimen by crushing. The samples of 30 g were collected from them and were vacuum-dried for 24 h. The pore size distribution test was performed using a mercury intrusion porosimetry (Poremaster 60GT, Quantachrime). For each mortar, the sample of the pore size distribution ranging from 0.007 to about 200 μm of pore diameter was measured.

3. RESULTS AND DISCUSSION

The compressive strengths at the age of 7, 28,

Table 3 Compressive strengths

Specimen	7 days (N/mm^2)	28 days (N/mm^2)	91 days (N/mm^2)
CWA40-0	51.1	63.3	77.1
CWA40-20	54.8	68.0	82.9
CWA40-40	52.7	68.1	80.0
CWA50-0	35.9	48.2	59.0
CWA50-20	30.8	49.5	65.5
CWA50-40	35.6	56.1	62.4
CWA60-0	22.6	35.5	54.6
CWA60-20	26.2	41.5	57.9
CWA60-40	22.7	40.5	55.2

and 91 days, which were presented in the previous study [20], are shown in Table 3. In each W/B, except for 7 days curing, the compressive strength of the CWA mortars containing the GGBS was higher than that without the GGBS.

3.1 Chloride Concentration Profile

The chloride concentration profiles for each W/B at 48 and 96 weeks are shown in Fig. 1 and Fig. 2, respectively. The chloride concentration profiles at 48 weeks were shown in [20]. The chloride ion penetration depth was decreased with decreasing the W/B and increasing the GGBS replacement level at each immersion time. In contrast, the chloride concentration near the exposed surface decreased at 96 weeks when compared with that at 48 weeks.

3.2 Apparent Chloride Diffusion Coefficient

The apparent chloride diffusion coefficient was determined by fitting the chloride concentration profile shown in Fig. 1 and 2 to the following Fick's second low,

$$C(x,t) = C_0\left(1 - erf\,\frac{x}{2\sqrt{D_a \cdot t}}\right) \quad (1)$$

where $C(x, t)$ is the chloride concentration (kg/m^3) at depth x (cm) and exposure time t (year), C_0 is the surface chloride concentration (kg/m^3), D_a is the apparent chloride diffusion coefficient (cm^2/year), and *erf* is the error function.

In the analysis of the chloride concentration profile, for the surface chloride concentration, the maximum value obtained from the result of EPMA was used and the apparent chloride diffusion coefficient was obtained by the curve fitting. The apparent chloride diffusion coefficients after analyzed are given in Table 4. The relationships between the apparent chloride diffusion coefficient and the GGBS replacement level for each W/B are shown in Fig. 3. The apparent chloride diffusion coefficient has a linear trend with the GGBS replacement level and the changing of the apparent chloride diffusion coefficients except for the CWA

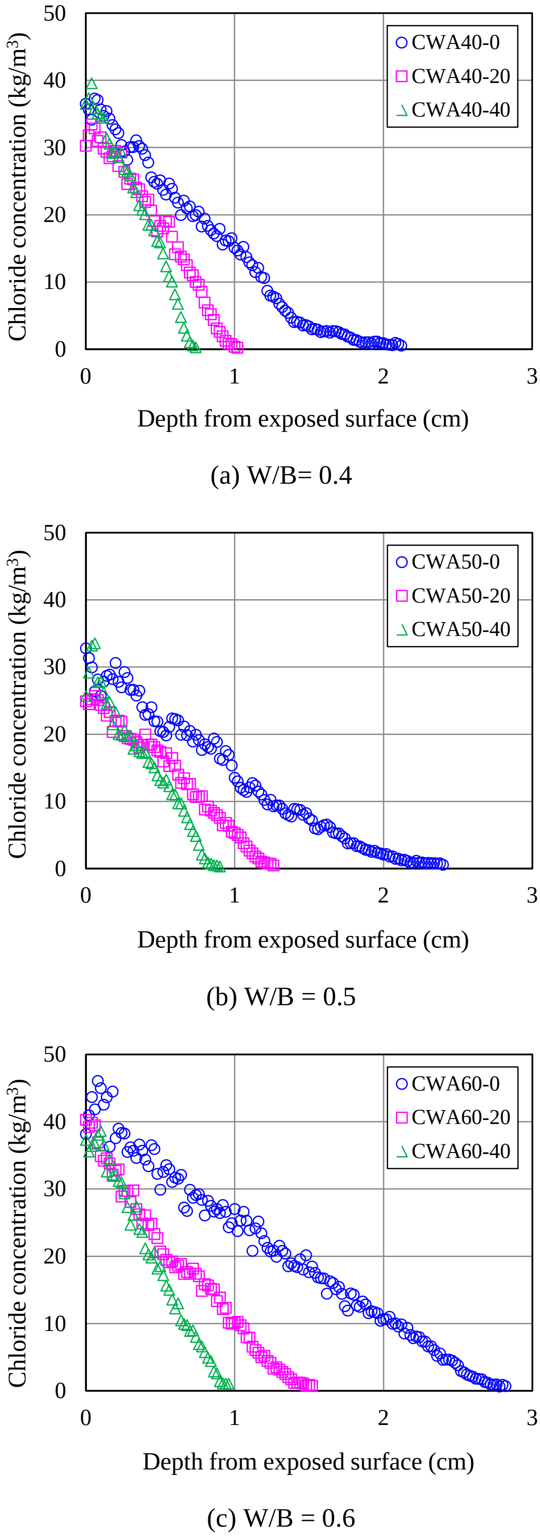
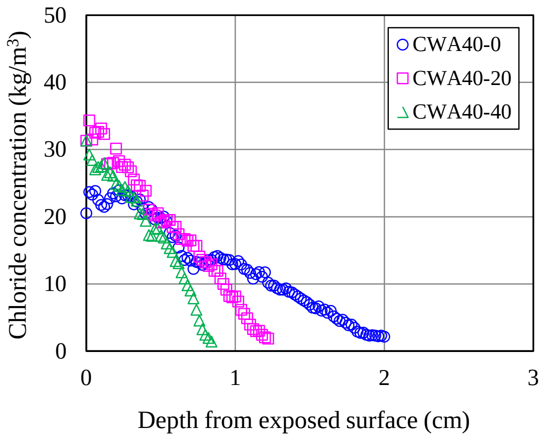
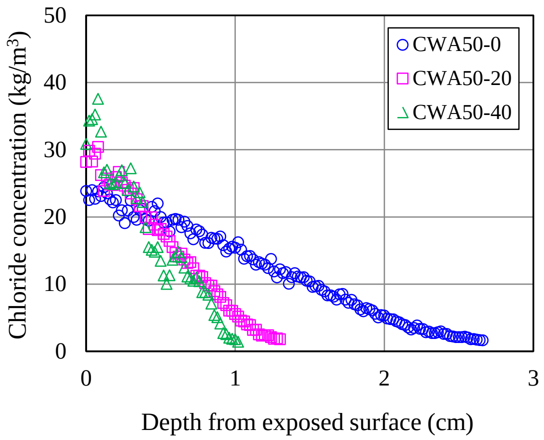


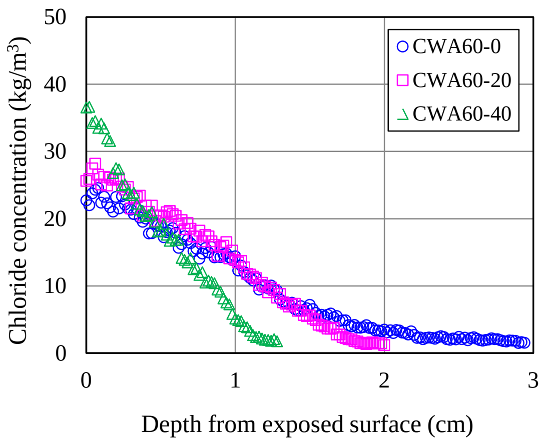
Fig. 1 Chloride concentration profiles at 48 weeks immersion [20]



(a) W/B = 0.4

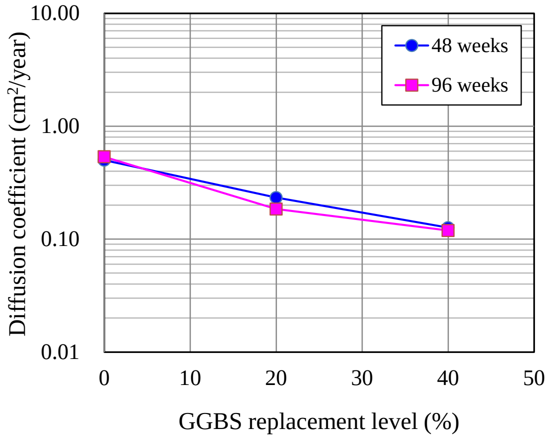


(b) W/B = 0.5

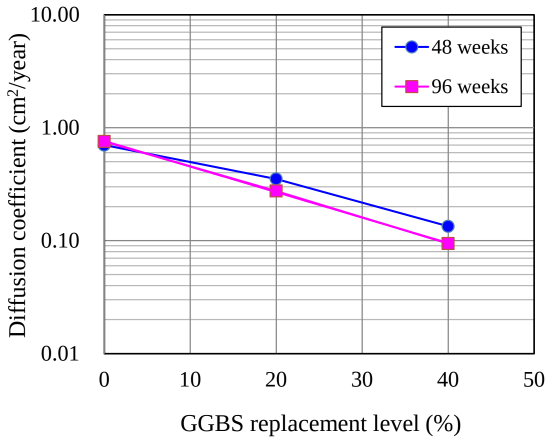


(c) = W/B = 0.6

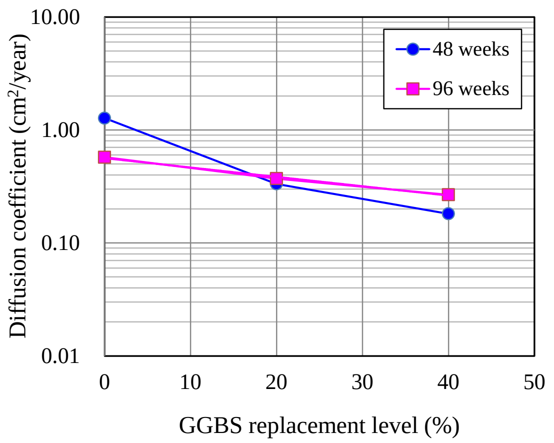
Fig. 2 Chloride concentration profiles at 96 weeks immersion



(a) W/B = 0.4



(b) W/B = 0.5



(c) = W/B = 0.6

Fig. 3 Relationships between apparent chloride diffusion coefficient and GGBS replacement level

Table 4 Apparent chloride diffusion coefficients

Specimen	48 weeks (cm^2/year)	96 weeks (cm^2/year)	Average (cm^2/year)
CWA40-0	0.500	0.535	0.518
CWA40-20	0.233	0.185	0.209
CWA40-40	0.126	0.119	0.123
CWA50-0	0.700	0.754	0.727
CWA50-20	0.351	0.275	0.313
CWA50-40	0.134	0.094	0.114
CWA60-0	1.271	0.573	0.922
CWA60-20	0.333	0.371	0.352
CWA60-40	0.181	0.267	0.224

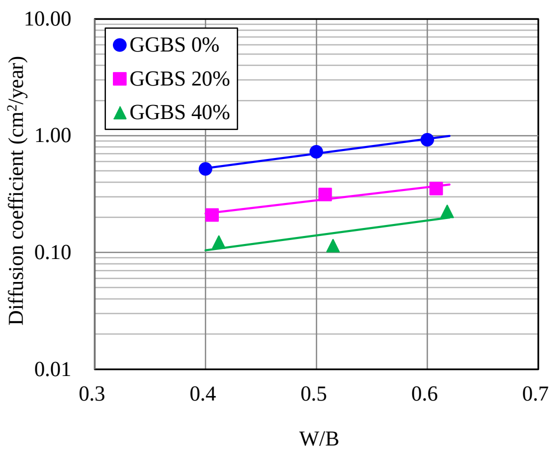


Fig. 4 Relationship between average apparent chloride diffusion coefficient and W/B

without the GGBS at the W/B of 0.6 was relatively small along the immersion time. The relationship between the average apparent chloride diffusion coefficient, which is averaged value of the apparent chloride diffusion coefficient at 48 and 96 weeks immersion shown in Table 4, and W/B is shown in Fig. 4. The average apparent chloride diffusion coefficient has a linear trend with the W/B. This is the same trend presented in the standard specifications for concrete structures, JSCE [21].

3.3 Pore Size Distribution

The pore size distributions ranging from 0.007 to about 200 μm diameter for each W/B at 48 and 96 weeks are shown in Fig. 5 and 6, respectively. The pore size distributions at 48 weeks were shown in [20]. At both immersion times, the pore volume was clearly decreased in the region of the smaller pore size when the W/B was lower and the distribution significantly changed in the smaller pore size less than around 0.1-0.2 μm. Furthermore, one peak value of the pore volume was observed in the CWA mortars without and with the GGBS of 20%. In contrast, in the CWA mortar with the GGBS of 40%, no peak value exhibited and the smaller pore size mostly occupied the pore volume when compared with the other specimens. Moon et al. [22] reported that mineral admixtures such as GGBS and fly ash reduce large pores in cement-based material with a diameter exceeding 0.05 mm such as macro pore. At 96 weeks, the pore size at the peak in the CWA without the GGBS was

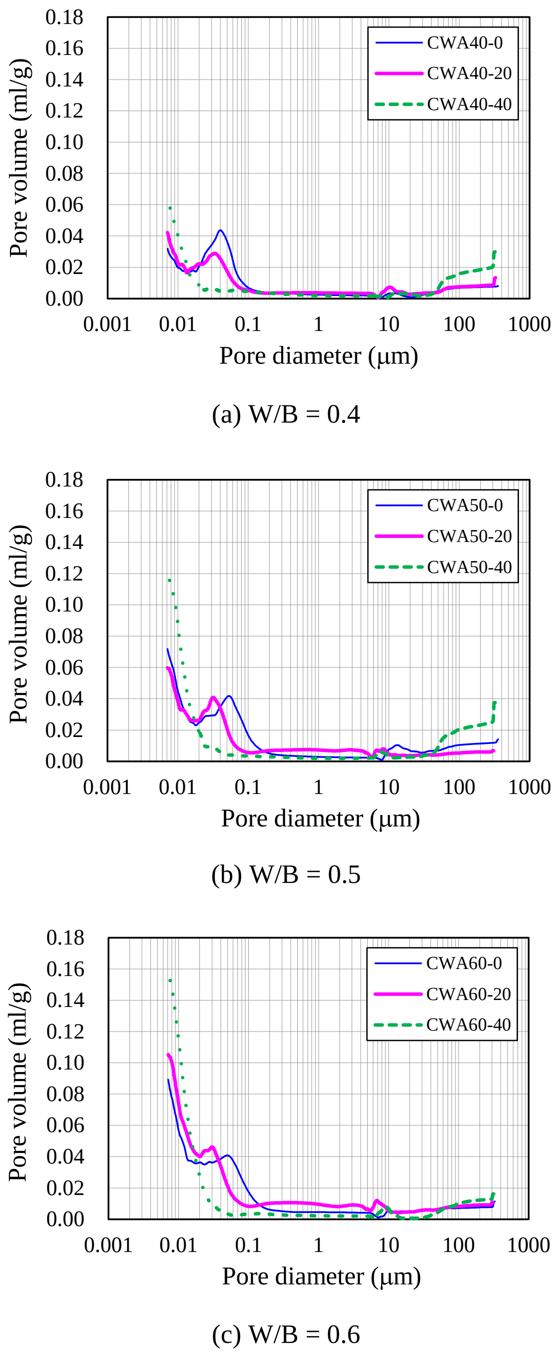
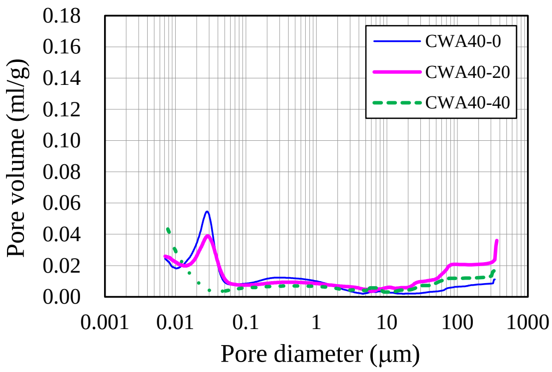
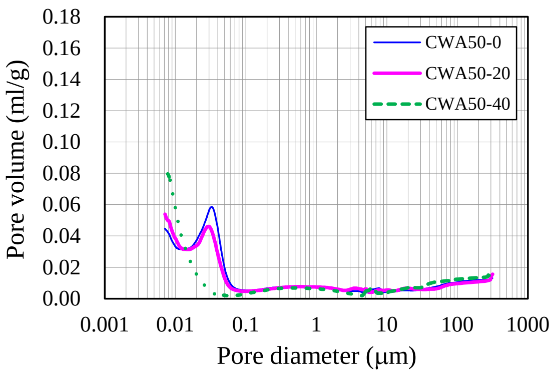


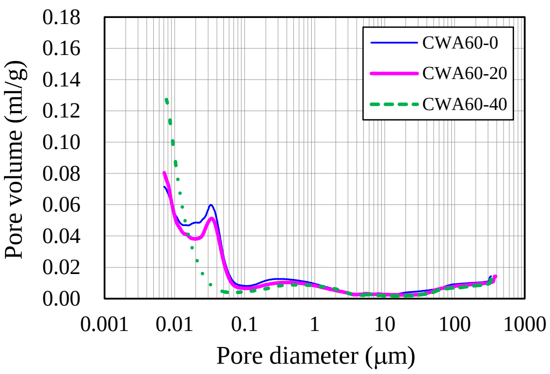
Fig. 5 Pore size distributions at 48 weeks immersion [20]



(a) W/B = 0.4



(b) W/B = 0.5



(c) W/B = 0.6

Fig. 6 Pore size distributions at 96 weeks immersion

shifted to smaller pore size when compared with that at 48 weeks.

3.4 Relationship between Apparent Chloride Diffusion Coefficient and Pore Volume

From the pore size distributions shown in Fig. 5 and 6, the relationship between the apparent chloride diffusion coefficient and the cumulative pore volume at 48 and 96 weeks was evaluated. In this study, the cumulative pore volume of each specimen was considered ranging from 0.007 to 0.2 μm and from 0.007 to 0.1 μm based on the above mentioned properties in the pore size distributions. Then, as shown in Fig. 7, the cumulative pore volume ranging from 0.007 to 0.1 μm was better correlation with the apparent chloride diffusion coefficient. The GGBS contributes to the more refined pore structure of the hydrated cementitious material and has the binding adsorption capacity [23]. Therefore, further investigation for a longer immersion time might be needed to understand its relationship.

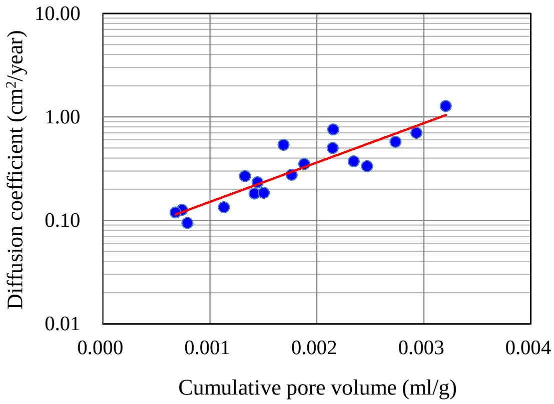


Fig. 7 Relationship between apparent chloride diffusion coefficient and cumulative pore volume

4. CONCLUSIONS

In this study, the apparent chloride diffusion coefficient and the pore size distribution of CWA mortars containing the GGBS at 48 and 96 weeks immersion were investigated. The following conclusions can be drawn.

(1) Regardless of the chloride immersion period, the use of GGBS significantly reduces the chloride diffusion in the CWA mortar. The reduction of the apparent chloride diffusion coefficient is proportional to the GGBS replacement level. Except for the CWA mortar at the W/B of 0.6 without any GGBS, the extended period of chloride immersion from 48 to 96 weeks has minimal effect on the apparent chloride diffusion coefficient.

(2) Based on the pore size distribution results, the pore volume especially in the range of pore diameter under 0.1-0.2 μm was clearly decreased when the W/B ratio was lowered. In addition, when the W/B was decreased, the apparent chloride diffusion coefficient was reduced. By correlating the cumulative pore volume of the pore diameter under 0.1 μm with the apparent chloride diffusion coefficient, a good correlation was found. Therefore, the cumulative pore volume plays

an important role to control the chloride diffusion.

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