

Chapter 4

Experimental Programs

4.1 General

The experimental program was designed to study influence of various parameters including mix proportion, type of fly ash, and conditions of the natural environments on carbonation of concrete and mortar. The conditions of the test were varied by the carbon dioxide concentration and raining subjection in the environments. In addition to the tests in natural environments, carbonation tests for concrete and mortar in accelerated environments were also conducted. The effect of curing duration, environmental relative humidity and temperature on carbonation resistance of mortar was examined. The relationship between carbonation depths of concrete exposed in natural and accelerated environments was established, which is beneficially used for predicting the carbonation depth in a natural environment.

4.2 Materials

4.2.1 Cementitious materials

Ordinary Portland cement type I (Elephant brand), conforming to ASTM C150 type I, and two samples of Lignite fly ashes, collected from Mae-Moh power plant, were used throughout the study. The two fly ashes, FA1 and FA2, have different chemical composition (mainly CaO content) but equivalent fineness. Class of fly ashes is classified by EIT 1014-46 as low-CaO and high-CaO for FA1 and FA2, respectively. However, both fly ashes are in the range of class C by ASTM C618. Chemical compositions and physical properties of the cementitious materials are shown in Table 4.1.

4.2.2 Aggregates

The fine aggregate was a natural river sand that passed sieve NO.4 and had a fineness modulus of 3.12. The coarse aggregate was a crushed limestone aggregate with a maximum size of 20 mm. The aggregate complied with the requirement of ASTM C33. The values of specific gravity of fine aggregate tested in accordance with ASTM C128 were 2.57 and 2.59 in dry and saturated-surface dry conditions, respectively. Specific gravity of coarse aggregate tested in accordance with ASTM C127 was 2.68 and 2.69 in dry and saturated-surface dry conditions, respectively.

Compacted void content of binary mixture of aggregates was performed according to ASTM C29/C29M-91a. Fig. 4.1 shows a void curve of binary mixture of aggregate and sand to total aggregate ratio (by volume). The sand to total aggregate ratio of 0.45, which gives a minimum void ratio of 0.23, was selected for use in all mix proportions of concrete. It is noted that a void ratio of 0.36 that is given by the sand to total aggregate ratio of 1.0 was used for the mix proportioning of mortars

Table 4.1 Chemical composition and physical properties of cement and fly ashes

	Cement	FA1	FA2
Chemical composition (%)			
Silicon dioxide	20.99	45.88	33.13
Aluminum oxide	5.18	26.20	26.89
Iron oxide	3.20	10.94	11.96
Calcium oxide	64.63	8.28	15.07
Magnesium oxide	1.30	2.83	2.94
Sulfur trioxide	2.61	1.04	1.63
Insoluble residue	0.13	-	-
Sodium oxide	0.04	0.9	1.42
Potassium oxide	0.40	2.78	3.33
Titanium dioxide	0.25	0.51	0.52
Phosphorus pentaoxide	0.05	0.1	0.16
Free Lime	0.75	0.18	-
Gypsum Content	5.60	-	-
Physical Properties			
Specific gravity (g/cm^3)	3.15	2.03	2.13
Loss of ignition (%)	1.17	0.17	0.04
Blaine Fineness (cm^2/g)	3150	3460	3430
Water requirement (%)	100	95.6	90.2

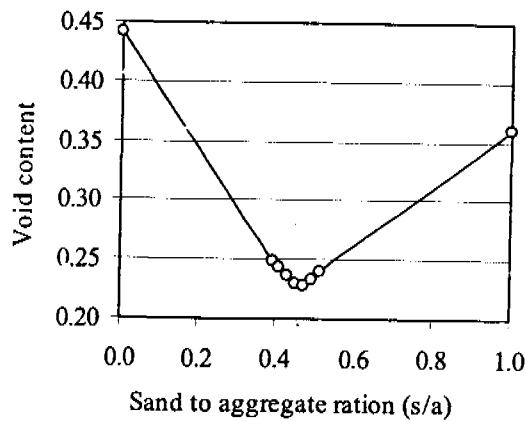


Fig. 4.1 Relationship between s/a (by volume) and void content

4.3 Carbonation Test in Real Environments

The main objective of this experiment is to examine the influences of mix proportion, type of fly ash, and environments on carbonation depth of fly ash concrete and mortar under real environments in Thailand. To investigate the effect of partial replacement of OPC with fly ash, plain OPC concrete mixtures that have water to binder ratio of 0.4, 0.5, and 0.6 and have γ (ratio of paste volume to the void volume of compacted

aggregate phase) of 1.2 and 1.4 were selected as reference mixtures. The replacement percentages of the two fly ashes were 10, 30, or 50%. For each concrete mixture, slump test was conducted within 15 minutes after mixing of concrete and then 10×20 cm cylinder and 10 cm cube specimens were cast for the test of 28-day compressive strength and the carbonation depth, respectively. The mix proportions and the slump results of the tested concrete mixtures were summarized in Table 4.2. The ratio of paste volume to void content of compacted aggregate phase (γ) is defined as follow.

$$\gamma = \frac{V_p}{V_{\text{void}}} \quad (4.1)$$

where V_p is the volume of paste in a unit volume of fresh concrete (m^3) and V_{void} is the volume of the void in the densely compacted total aggregate (fine and coarse aggregate) in a unit bulk volume of the aggregate (m^3). The volume of paste can be derived as,

$$V_p = V_c + V_f + V_w + V_{\text{air}} \quad (4.2)$$

where V_c , V_w , V_{air} and V_{pow} are the volume of cement, fly ash, water, and air, respectively, in a unit volume of concrete mixture (m^3).

Table 4.2 Mix proportion of concrete in natural environment

No.	γ	w/b	%r	Cement (kg/m^3)	FA (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)	Gravel (kg/m^3)	SL (cm)
Concrete made with FA1									
c-1	1.2	0.50	0	325	0	163	1071	844	1.0
c-2	1.2	0.50	10	287	32	159	1071	844	1.0
c-3	1.2	0.50	30	214	92	153	1071	844	1.5
c-4	1.2	0.50	50	147	147	147	1071	844	1.5
c-5	1.2	0.60	0	290	0	174	1071	844	5.5
c-6	1.2	0.60	10	256	28	171	1071	844	5.0
c-7	1.2	0.60	30	192	82	165	1071	844	4.5
c-8	1.2	0.60	50	132	132	159	1071	844	7.0
c-9	1.4	0.40	0	435	0	174	1003	790	2.0
c-10	1.4	0.40	10	382	42	170	1003	790	2.0
c-11	1.4	0.40	30	284	122	162	1003	790	3.0
c-12	1.4	0.40	50	194	194	155	1003	790	3.5
c-13	1.4	0.50	0	382	0	191	1003	790	12.0
c-14	1.4	0.50	10	336	37	187	1003	790	12.5
c-15	1.4	0.50	30	251	108	179	1003	790	14.0
c-16	1.4	0.50	50	172	172	172	1003	790	15.0
c-17	1.4	0.60	0	340	0	204	1003	790	18.0
c-18	1.4	0.60	10	300	33	200	1003	790	19.0
c-19	1.4	0.60	30	225	96	193	1003	790	20.0

Concrete made with FA2									
c-20	1.4	0.60	50	155	155	186	1003	790	20.0
c-21	1.2	0.50	0	325	0	163	1071	844	1.0
c-22	1.2	0.50	10	288	32	160	1071	844	1.0
c-23	1.2	0.50	30	216	92	154	1071	844	2.0
c-24	1.2	0.50	50	149	149	149	1071	844	2.0
c-25	1.2	0.60	0	290	0	174	1071	844	5.5
c-26	1.2	0.60	10	257	29	171	1071	844	5.5
c-27	1.2	0.60	30	193	83	166	1071	844	5.5
c-28	1.2	0.60	50	134	134	161	1071	844	8.5
c-29	1.4	0.40	0	435	0	174	1003	790	2.0
c-30	1.4	0.40	10	383	43	170	1003	790	6.0
c-31	1.4	0.40	30	286	123	164	1003	790	6.5
c-32	1.4	0.40	50	197	197	157	1003	790	5.0
c-33	1.4	0.50	0	382	0	191	1003	790	12.0
c-34	1.4	0.50	10	337	37	187	1003	790	13.0
c-35	1.4	0.50	30	253	108	181	1003	790	13.0
c-36	1.4	0.50	50	175	175	175	1003	790	14.5
c-37	1.4	0.60	0	340	0	204	1003	790	18.0
c-38	1.4	0.60	10	301	33	201	1003	790	18.5
c-39	1.4	0.60	30	227	97	194	1003	790	19.0
c-40	1.4	0.60	50	157	157	188	1003	790	19.0

Table 4.3 Mix proportion of mortar in natural environment

No.	γ	w/b	%r	Cement (kg/m ³)	FA (kg/m ³)	Water (kg/m ³)	Sand (kg/m ³)
Mortar made with FA1							
M1	1.2	0.50	0	504	0	252	1488
M2	1.2	0.50	10	443	49	247	1488
M3	1.2	0.50	30	329	141	238	1488
M4	1.2	0.50	50	225	225	230	1488
M5	1.4	0.40	0	675	0	270	1300
M6	1.4	0.40	10	591	66	264	1300
M7	1.4	0.40	30	437	187	253	1300
M8	1.4	0.40	50	297	297	243	1300
M9	1.4	0.50	0	592	0	296	1300
M10	1.4	0.50	10	520	58	290	1300
M11	1.4	0.50	30	386	165	280	1300
M12	1.4	0.50	50	264	264	270	1300

Mortar made with FA2							
M13	1.2	0.50	0	504	0	252	1488
M14	1.2	0.50	10	445	49	247	1488
M15	1.2	0.50	30	334	143	239	1488
M16	1.2	0.50	50	231	231	231	1488
M17	1.4	0.40	0	675	0	270	1300
M18	1.4	0.40	10	595	66	264	1300
M19	1.4	0.40	30	444	190	254	1300
M20	1.4	0.40	50	305	305	244	1300
M21	1.4	0.50	0	592	0	296	1300
M22	1.4	0.50	10	523	58	291	1300
M23	1.4	0.50	30	393	168	280	1300
M24	1.4	0.50	50	271	271	271	1300

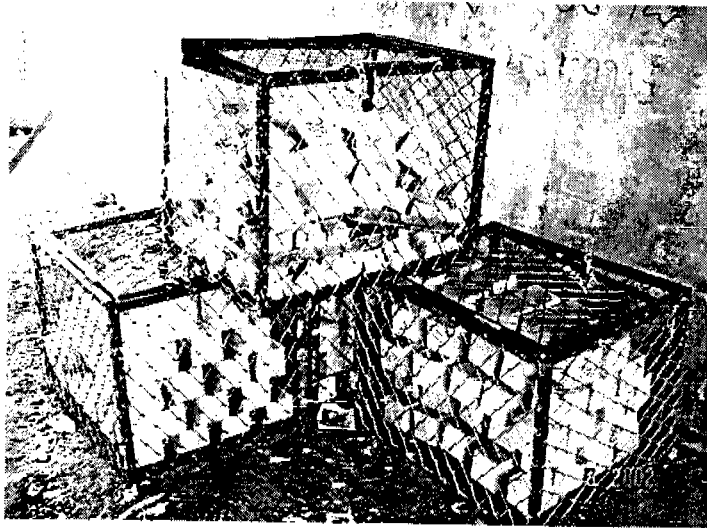


Fig. 4.2 Concrete specimens in the city-sheltered conditions

In addition to the concrete test, mortar specimens were also cast in 5 cm cubes. Twenty-four mixtures of mortar were proportioned by varying the value of γ , water to binder ratio, type and replacement percentage of fly ash. The mix proportions of mortar were summarized in Table 4.3. All concrete and mortar specimens were cured for 28 days in water before being exposed to real environments for the carbonation experiment for periods of 3, 6, 12, 18, and 24 months. Three locations were selected to examine the influences of rain subjection and carbon dioxide concentration as in the following.

1. The city of Bangkok. This area had the highest carbon dioxide concentration. There are two exposing conditions in this location, i.e. condition with and without possibility of rain subjection (city-non-sheltered and city-sheltered). All concrete and mortar specimens were exposed in the city-sheltered condition (see Fig. 4.2). Additional concrete specimens (C1 to C4 and C13 to C16) were cast for the exposure in the city-non-sheltered condition.

2. The rural area outside Bangkok (rural environment). The specimens were stored in the laboratory building of the Sirindhorn International Institute of Technology, Thammasat University and kept away from rain. Concrete specimens with mix proportions C1 to C8 were cast for the exposure in this location.

3. At a location closed to the sea in Chonburi Province (seaside environment). The specimens were kept away from rain. Concrete specimens with mix proportions C1 to C8 were cast for the exposure in this location. The conditions of the environments are shown in Table 4.4.

Table 4.4 Environmental conditions (annual average)

	City environment	Rural environment	Seaside environment
RH (%) ^a	72.5 (sheltered), 83.0 (non-sheltered)	80.0	72.0
Temperature (°C) ^a	28.9	28.1	28.7
CO ₂ concentration (ppm) ^b	625	300	225

Note: * obtained from the Meteorological department

** measured by the author

At the age of measurement, specimens were tested for carbonation depth in the laboratory. The specimens were split and cleaned. The depths of carbonation were determined by spraying on a freshly broken surface with 1% of phenolphthalein in the solution of 70% ethyl alcohol (RILEM 1988). The phenolphthalein solution is colorless and used as an acid-base indicator. The color of the solution changes into purple when pH is higher than the range of approximately nine. Therefore, when the solution is sprayed on a broken concrete surface, the carbonated portion is uncolored (concrete color) and non-carbonated portion is purple (see Fig. 4.3 for concrete specimens and Fig. 4.4 for mortar specimens). The depth of carbonation is defined as the thickness of carbonated portion. The average depths of carbonation were measured and reported.



Fig. 4.3 Example of carbonated concrete specimens after being tested

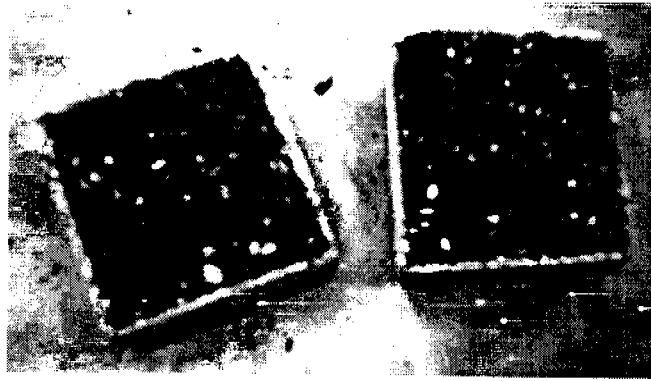


Fig. 4.4 Example of carbonated mortar specimens after being tested

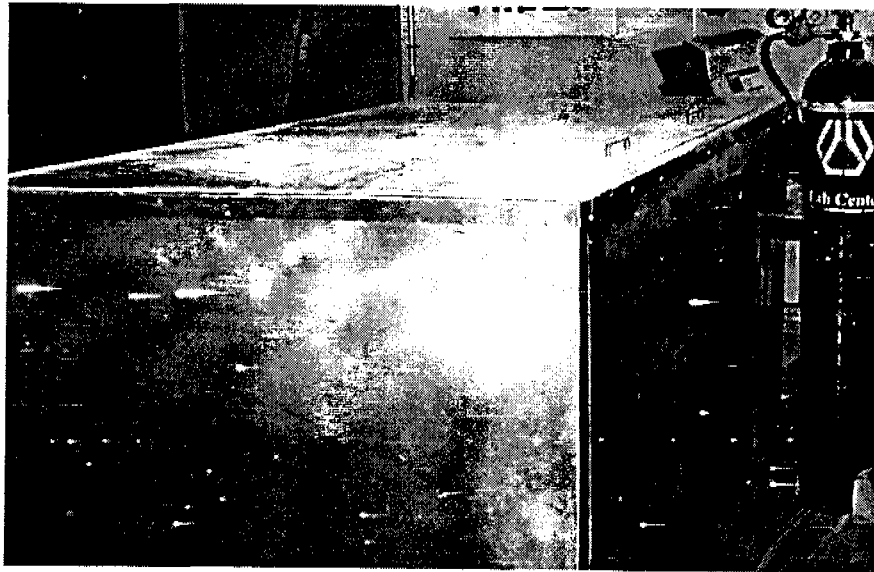


Fig. 4.5 Accelerated carbonation chamber

4.4 Carbonation Tests of Concrete in Accelerated Environment

The rate of carbonation in real environment is usually slow due to low CO_2 concentration level in the environment. Hence, the accelerated carbonation test is a practical method used to assess the carbonation resistance of concrete within a reasonably short time. It is practically beneficial to estimate the carbonation depth in real environment based on the results of the accelerated test. Some of the concrete mixtures designed for the exposure in real environments were selected for the accelerated carbonation test (C1 to C3, C5 to C7, C13 to C15, C25, and C26). The concrete specimens were also moist-cured for 28 days before being carbonated in the accelerated carbonation chamber invented in this study (see Fig. 4.5) for a period of 4 and 8 weeks. The temperature and relative humidity in the carbonation chamber were controlled at 40°C and $55 \pm 5\%$, respectively. The carbon dioxide concentration was 4% (40,000 ppm). At the age of measurement, specimens were tested for carbonation depth by the same procedure as those tested in the natural environment exposure test.

4.5 Carbonation Tests of Mortar in Accelerated Environments

The main objective of this experiment is to examine the influence of curing duration, environmental temperature, and environmental relative humidity on carbonation depth of fly ash mortar under controlled accelerated carbonation environment. To investigate the effect of partial replacement of OPC with fly ash, plain OPC mortar mixtures having w/b of 0.3 and 0.5 were selected as reference mixtures (aggregate to binder ratio, a/b of 1.0 and 2.0, respectively). The replacement percentages of fly ash were 0, 10, or 30%. For each mixture, 50 mm cubes specimens were cast and demolded at 24 hours after casting. The specimens were cured in water for 7, 14, and 28 days. The mixture proportions of mortar for accelerated carbonation test are summarized in Table 4.5. There are three experiments for these mortar specimens as follows.

Table 4.5 Mix proportion of mortar in accelerated environments

No.	Fly ash	w/b	A/B	%r	Cement (kg/m ³)	FA (kg/m ³)	Water (kg/m ³)	Sand (kg/m ³)
MA1	-	0.3	1:2	0	980	0	294	980
MA2	1	0.3	1:2	10	868	96	289	964
MA3	1	0.3	1:2	30	652	280	280	932
MA4	2	0.3	1:2	10	869	97	290	966
MA5	2	0.3	1:2	30	654	280	280	934
MA6	-	0.5	1:1	0	553	0	277	1383
MA7	1	0.5	1:1	10	493	55	274	1370
MA8	1	0.5	1:1	30	376	161	269	1343
MA9	2	0.5	1:1	10	493	55	274	1370
MA10	2	0.5	1:1	30	376	161	269	1343

4.5.1 Compressive strength

After the specimens were cured for a period of 7, 14, or 28 days, they were tested for compressive strength. The test procedure was in accordance with ASTM C39. The average compressive strength of three specimens was reported.

4.5.2 Total Permeable void

The total permeable void was measured to study the effect of fly ash on the total porosity of mortar. The test procedure was in accordance with ASTM C642. After being water-cured for 28 days, the specimens were dried in an oven at a temperature of 105 °C for 24 hours. Then, the oven-dried mass was determined (W_1). After that, specimens were boiled for five hours to saturate the specimens and to evacuate the entrapped air bubbles within the specimens. The saturated surface-dried mass in air (W_2) and apparent mass in water (W_3) were determined. The volume of permeable voids was calculated as follow.

$$V = \frac{W_2 - W_1}{W_2 - W_3} \times 100 \quad (4.3)$$

where V is the amount of permeable voids (%).

4.5.3 Carbonation test

After the specimens were cured for a period of 7, 14, or 28 days, they were stored in the accelerated carbonation chamber (see Fig. 4.4) for a period of 4 and 8 weeks. The environment in the chamber had a CO₂ concentration of 4% (40,000 ppm). Temperature and relative humidity were controlled at 40°C and 55 ± 5%, respectively. In addition to this condition, the tests under the conditions that have temperature and relative humidity of 40°C and 40 ± 5%, respectively and 30°C and 60 ± 5, respectively were conducted for the mortar specimens cured for 28 days. The effect of environmental condition was then examined. The carbon dioxide concentration was maintained constant for both conditions at 4% (40,000 ppm).

4.6 Relative Water Content Test

Relative water content within concrete pores is one of the most important parameters affecting the ingress of carbon dioxide in concrete. Because concrete is an originally porous material, it always contains some water within its pores in a normal environment. When pores are filled with water, the gas diffusion ceases and the concrete does not undergo significant carbonation. However, when concrete is subjected to drying, water in the concrete pores evaporates and thus carbon dioxide is able to penetrate into the concrete. The concrete can undergo significant carbonation. In this section, the experiment was designed to study the migration of moisture from concrete. The relative water contents in concrete after being subjected to drying were investigated. The relative water content is defined as follow. (Tangtermsirikul et al 1992)

$$C_w = 100 \times \frac{W_e}{W_{sat}} \quad (4.4)$$

where C_w is the relative water content (%). W_e is the amount of evaporable water in the air-dried concrete after being subjected to drying (kg). W_{sat} is the amount of evaporable water in the water saturated concrete (kg).

The mortars were proportioned by varying water to binder ratio, fly ash content (FA2), and aggregate content. The mix proportions are shown in Table 4.6. In the series of mortar with water to binder ratio of 0.4, sand was replaced by gravel in order to examine to effect of type of aggregate (mixes MW10, MW11, and MW12). The specimens were cast in cylinders that have a diameter and length of 2.54 cm and 10 cm, respectively. The specimens were cured for 28 day in water. After curing, the specimens were sealed at the bottom to allow one-way evaporation at the top and stored in a condition of 40 °C with 50 ± 5 %RH until an age of 3, 7, or 28 days. In addition to this condition, the effect of drying condition was studied from the experiment on paste specimens. The paste specimens had water to binder ratio of 0.4 and fly ash content (FA2) of 0, 20, or 60%. The paste specimens were water-cured and dried for 28 days. They were tested under the conditions of 30 °C with 65 ± 5 %RH and 40 °C with 50 ± 5 %RH.

In determining the evaporable water content at the age of measurement, the specimens were cut to have the thickness of 1 cm per slice from the open surface to the distance of 4 cm from that surface (see Fig. 4.6). The splitting process was selected in order to prevent water loss during cutting. The cut specimens were oven-dried at 105 °C for 24 hours to determine the weight loss. This weight loss represents the amount of

evaporable water (W_e). The evaporable water content of the water-saturated concrete (W_{sat}) was determined by submerging the oven-dried specimens in water for 24 hours to determine the weight gain at the saturated-surface dried condition.

Table 4.6 Mix proportion of mixtures for relative water content test

No.	w/b	%r	A/B	Cement (kg/m ³)	FA (kg/m ³)	Water (kg/m ³)	Sand (kg/in ²)	Gravel (kg/m ³)
MW1	0.3	0	1.0	978	0	293	978	-
MW2	0.3	20	1.0	755	189	283	944	-
MW3	0.3	60	1.0	353	529	264	882	-
MW4	0.4	0	2.0	659	0	264	1318	-
MW5	0.4	20	2.0	515	129	257	1287	-
MW6	0.4	60	2.0	246	368	246	1228	-
MW7	0.5	0	3.0	497	0	249	1491	-
MW8	0.5	20	3.0	391	98	244	1464	-
MW9	0.5	60	3.0	188	283	235	1413	-
MW10	0.4	0	2.0	672	0	269	-	1344
MW11	0.4	20	2.0	525	131	262	-	1311
MW12	0.4	60	2.0	250	375	250	-	1250

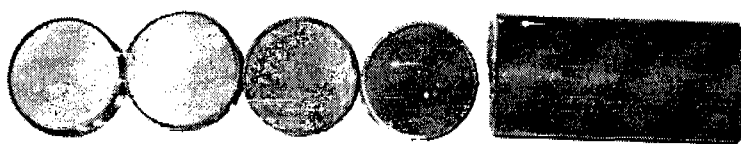


Fig. 4.6 Example of mortar specimens after being spitted