

COMPRESSIVE STRENGTH AND ACID RESISTANCE CHARACTERISTICS OF CEMENT PASTE AND MORTAR INCORPORATING WASTE CERAMIC INSULATOR

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ABSTRACT: A series of cement paste and mortar specimens containing waste ceramic insulator and blast-furnace slag was made. The compressive strength and the acid resistance of these materials are investigated here. Among 18 specimens of cement paste, only two showed a compressive strength less than the standard specified for the sulfuric acid-proof mortar. These tests show that the waste ceramic insulator powder can be added by approximately 0.4 parts by weight in the powder materials of cement paste, and the waste ceramic insulator aggregate can be added by two-thirds by weight in the materials of mortar. The acid resistance was tested by immersing the specimens in 5% sulfuric acid. The reaction layer formation of specimens is affected by the immersion time, temperature, and compounding ratio of the materials. The results show that adding the waste ceramic insulator controls the dilatability of the reaction layer of cement paste and mortar.

Keywords: Ceramic Insulator, Cement Paste, Mortar; Compressive Strength, Acid Resistance

1. INTRODUCTION

Ceramic insulators are used between high voltage electric wires and their supporting poles or towers. The insulating property of the ceramic is lost if it is cracked with age and deterioration or by lightning, and such insulators require regular replacement. An electric power company can be one of the main facilities that discharge ceramic waste. In our survey on major electric power companies in Japan, the amount of generated ceramic waste exceeds 10,000 tons each year, and approximately 40 % of the waste ceramic insulators are disposed of at landfill sites. Some ceramic waste is incorporated into blocks, tiles, and other building materials. However, this ceramic material needs other ways of recycling.

The present study investigates materials used in the manufacture of sewer pipes as potential material to which ceramic waste can be added. Concrete corrosion by sulfuric acid has been a major deterioration affecting sewer systems in the world [1]-[5]. The annual cost of arising from corrosion of concrete used in the water and wastewater infrastructure has been estimated to be about US\$ 36 billion in the USA [6]. The sulfuric acid ions contained in sewage are changed into hydrogen sulfide by sulfate reducing bacteria. Hydrogen sulfide becomes gas-like and serves as sulfuric acid with the sulfur oxidation bacteria and fungi present in the sewer systems [7]-[9]. Thus, the generated sulfuric acid accelerates corrosion of the concrete structure. Therefore, the rehabilitation and replacement of corrosion-damaged sewers are an

urgent issue.

Some researchers have studied acid-resistant concretes [10]-[13], but such concretes are not always satisfactory due to their problems with durability. Several studies show that concretes with added blast-furnace slag have increased acid resistance [14], [15]. However, it was reported that the expansion pressure inside the concrete can cause surface exfoliation depending on the proportion of blast-furnace slag in the material [16]. The acid resistance of concrete with added blast-furnace slag still requires improvement.

The present study aims to develop a concrete with acid resistance and durability by adding ceramic waste to a cement matrix containing conventional blast-furnace slag. There are many studies on ceramic waste added to a cement matrix [17]-[26], but few studies discuss the resistance of a cement complex against sulfuric acid in sewer systems. Cement pastes and mortar, with various proportions of blast-furnace slag, and ceramic waste were made and, the compressive strength of each test material was investigated. Moreover, the acid resistance of each test material was examined by comparing immersion times and temperatures. Some knowledge was acquired about the acid resistance of the cement paste and mortar containing ceramic waste and blast-furnace slag.

2. METHODS

2.1 Materials

Specimens of cement paste and mortar were

made from municipal tap water, ordinary Portland cement (SUMITOMO OSAKA CEMENT Co., LTD., Japan), blast-furnace slag aggregate (SHINKO SLAG PRODUCTS Co., Ltd., Japan), blast-furnace slag powder (Nippon Steel & Sumikin Cement Co., Ltd., Japan), and waste ceramic insulator obtained from The Kanden L&A Company, Ltd. in Japan. Aggregates and powders of blast-furnace slag and waste ceramic insulator were used in the present study. The particle size is <2.5 mm for blast-furnace slag aggregate, 0.075–5 mm for waste ceramic insulator aggregate, and <0.075 mm for waste ceramic insulator powder. Their specific gravity, specific surface area and main component are given in Table 1. The main components are CaO and SiO₂ for ordinary Portland cement and blast-furnace slag. The main components are SiO₂ and Al₂O₃ for waste ceramic insulator.

Table 1. Physical properties and main components of materials.

	C	IA	IP	SA	SP
Specific gravity	3.15	2.40	2.50	2.73	2.91
Specific surface area (cm ² /g)	3366	-	3887	-	6230
Main components	CaO:64%	SiO ₂ :71%		CaO:42%	
	SiO ₂ :21%	Al ₂ O ₃ :21%		SiO ₂ :34%	
	Al ₂ O ₃ :5%	K ₂ O:4%		Al ₂ O ₃ :14%	
				MgO:7%	

C: cement; IA: waste ceramic insulator aggregate; IP: waste ceramic insulator powder; SA: blast-furnace slag aggregate; SP: blast-furnace slag powder.

2.2 Specimens

Cement paste specimens were made from water, ordinary Portland cement (C), waste ceramic insulator powder (IP), and blast-furnace slag powder (SP). The weight ratio of water to (C + IP + SP) was fixed to 0.5, and the proportions (g/g/g) of cement to SP to IP were varied for each specimen.

Mortar specimens were made from water, C, ceramic insulator aggregate (IA), IP, blast-furnace slag aggregate (SA), and SP. The weight ratio of water to (C + SP) was fixed to 0.5, and that of (IA + IP + SA) to (C + SP) was fixed to 2.0. The proportions (g/g/g/g) of cement to the other materials were varied for each specimen.

To prepare the cement paste, C, (IP + SP) and water were mixed with a Hobart mixer of 5 L capacity. To prepare the mortar, C, (IA + IP + SA), SP, and water were mixed with the Hobart mixer. IP was added into the mortar material as an aggregate mixture. The mixture was poured into a mold and left to stand for one day at room temperature. Then, it was taken out of the mold and

cured in water at 20°C for 27 days. All the specimens were pillar-shaped objects of 50 mm dia. x 100 mm height.

2.3 Compressive strength and acid resistance tests

The compression test was carried out after the specimens underwater were cured for 27 days. The diameter of each specimen was measured and compressed with a 500kN capacity universal testing machine (MARUI & Co., LTD., Japan) at a constant loading speed of 0.2 N/mm²/sec in accordance with JSCE-G 505 [27]. The load value at which the specimen destroyed was used as the maximum load. The compressive strength (N/mm²) was calculated as $P/(\pi(d/2)^2)$. Here, P is a maximum load (N) and d is a diameter of a specimen (mm).

In the acid resistance test, 5 % sulfuric acid (Wako Pure Chemical Industries, Ltd., Japan) was used. Each specimen was left for one day after the underwater curing for 27 days. After the diameter, height, and weight of each specimen were measured, the specimen was immersed in the sulfuric acid. The sulfuric acid was replaced every two weeks to maintain its pH. At various time points, the specimen was removed from the sulfuric acid, rinsed and immersed in municipal tap water for 24 hrs. The specimen was then allowed to dry for several days at room temperature, and the diameter, height, and weight of the specimen were measured. The specimen was cut with a concrete cutter (Ezaki Metal Co, LTD., Japan) and sprayed with a phenolphthalein solution. The phenolphthalein solution was prepared as follows; 1 g of phenolphthalein was dissolved to 90 mL of 95 % ethanol (both Wako Pure Chemical Industries, Ltd., Japan), and distilled deionized water was added up to 100 mL. The diameter of the pink colored region at the cutting plane, and the thickness of the reaction layer were measured (Fig. 1).

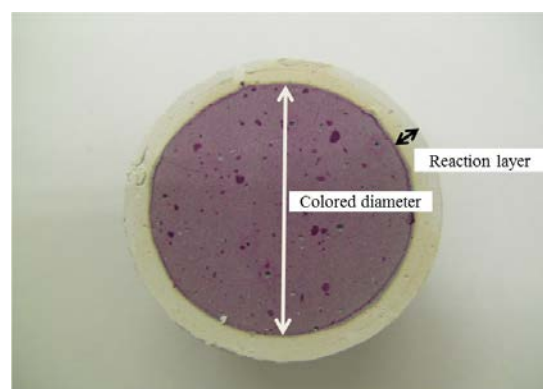


Fig. 1. Specimen sprayed with phenolphthalein solution after acid immersion

The pink colored region was not corroded by the sulfuric acid. The neutralization depth was calculated as half of the value by deducting the diameter of the pink colored region of the specimen from the diameter of the specimen before the immersion. The reaction layer surface was observed to analyze its composition using a scanning electron microscope (S-4800 Type II; Hitachi, Ltd., Japan).

3. RESULTS AND DISCUSSION

3.1 Influence of waste ceramic insulator and blast-furnace slag on compressive strength

The compressive strength measured for 18 types of cement paste is shown in Table 1. The highest arithmetic mean of the compressive strength was 49.6 N/mm² for the C:IP:SP ratio of 3:2:5, and the lowest arithmetic mean was 11.6 N/mm² for the C:IP:SP ratio of 4:6:0. The compressive strength for all specimens except those with the C:IP:SP ratios of 6:4:0 and 4:6:0 exceed 24 N/mm², which is the standard specified for sulfuric acid-proof mortar used for a corrosion-protective covering layer [28].

The relationship between parts of materials, or IP and SP, and the arithmetic means of the compressive strength is shown in Fig. 2. The compressive strength tends to be lower as the parts SP increase. A greater decrease in the compressive strength was observed as the parts IP increase. The average compressive strength for specimens with the C:IP:SP ratio of 6:4:0 was 20.2 N/mm². However, for those with the C:IP:SP ratios of 4:4:2 and 3:4:3, the average compressive strength was more than 24 N/mm². The tests of cement paste show that the waste ceramic insulator powder can be added by approximately 0.4 parts by weight in the powder materials of the cement paste containing blast-furnace slag.

The compressive strength for 15 types of mortar is shown in Table 2. The highest average of compressive strength was 65.1 N/mm² for the C:IA:IP:SA:SP ratio of 5:16:4:0:5, and the lowest was 50.3 N/mm² for the C:IA:IP:SA:SP ratio of 4:0:0:20:6.

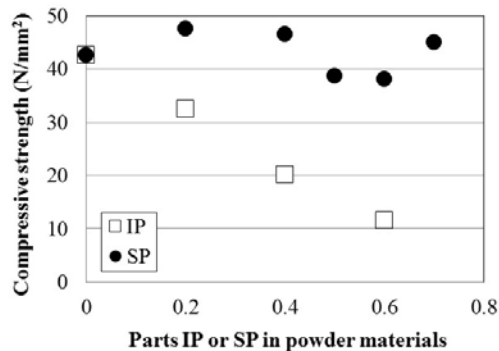


Fig. 2. Influence of waste ceramic insulator (IP) and blast-furnace slag (SP) on compressive strength

Table 1 Compressive strength for cement paste

Specimen proportion (C:IP:SP)	Compressive strength (N/mm ²)
10:0:0	42.5 ± 2.3 (6)
8:0:2	47.5 ± 7.0 (3)
8:2:0	32.5 ± 1.3 (3)
6:0:4	46.5 ± 5.4 (3)
6:4:0	20.2 ± 0.4 (3)
5:0:5	38.6 ± 1.8 (3)
5:1:4	41.0 ± 5.0 (6)
5:2:3	37.0 ± 0.8 (2)
5:3:2	37.3 ± 4.5 (6)
4:0:6	38.1 ± 3.5 (2)
4:1:5	40.5 ± 3.9 (3)
4:2:4	35.7 ± 1.8 (3)
4:3:3	34.5 ± 0.9 (3)
4:4:2	26.9 ± 0.0 (2)
4:6:0	11.6 ± 0.1 (3)
3:0:7	44.9 ± 5.4 (3)
3:2:5	49.6 ± 4.2 (3)
3:4:3	34.2 ± 2.1 (3)

C: cement; IP: waste ceramic insulator powder; SP: blast-furnace slag powder. Water/(C+IP+SP) = 0.5. The arithmetic mean ± standard error (range) is given. The number of specimens is given in parentheses.

Table 2 Compressive strength for mortar

Specimen proportion (C:IA:IP:SA:SP)	Compressive strength (N/mm ²)
10:20:0:0:0	54.3 ± 1.8 (3)
10:18:2:0:0	54.7 ± 1.4 (3)
10:16:4:0:0	60.2 ± 1.8 (3)
10:14:6:0:0	57.4 ± 2.9 (3)
5:20:0:0:5	58.6 ± 1.0 (3)
5:18:2:0:5	64.5 ± 3.5 (3)
5:16:4:0:5	65.1 ± 2.8 (3)
5:14:6:0:5	52.1 ± 14.3 (3)
4:20:0:0:6	51.9 ± 2.9 (3)
4:18:2:0:6	56.9 ± 2.0 (3)
4:16:4:0:6	63.7 ± 1.3 (3)
4:14:6:0:6	61.1 ± 0.8 (3)
4:0:0:20:6	50.3 ± 0.9 (3)
4:0:4:16:6	62.7 ± 2.4 (6)
4:8:4:8:6	56.9 ± 8.3 (6)

C: cement; IA: waste ceramic insulator aggregate; IP: waste ceramic insulator powder; SA: blast-furnace slag aggregate; SP: blast-furnace slag powder. Water/(C+SP) = 0.5 and (IA+IP+SA)/(C+SP) = 2.0. The arithmetic mean ± standard error (range) is given. The number of specimens is given in parentheses.

The average compressive strength for all ratios was more than 24 N/mm². The tests of mortar show that the waste ceramic insulator aggregate can be added by two-thirds by weight in the sum of powder and aggregate materials of the mortar.

3.2 Influence factors on formation of reaction layer in acidic solution

Results of acid resistance tests for the 23 cement pastes and the 44 mortars are shown in Tables 3 and 4. A white or gray reaction layer formed in all the specimens. The surface of some specimens was brown. The width of the reaction layer was 1.63–8.62 mm for the cement pastes and 0.48–10.15 mm for the mortars. The range of neutralization depths was 1.47–6.62 mm for the cement pastes and 1.35–13.66 mm for the mortars.

Table 3 Reaction layer and neutralization depth for cement paste in acid resistance tests

Specimen proportion (C:IP:SP)	T (°C)	IT (d)	RL (mm)	ND (mm)
10:0:0	18.4	30	1.83	1.52
5:4:1			2.25	1.70
5:2:3			2.63	2.24
3:7:0			2.43	1.48
3:5:2			3.12	2.23
3:3:4	21.1	60	3.93	3.01
4:6:0			5.13	2.90
4:2:4			8.62	6.62
4:6:0	22.4	30	2.53	1.47
4:5:1			3.08	1.70
4:4:2			3.18	2.00
4:3:3			3.90	2.99
4:2:4			4.53	3.47
8:2:0	23.5	30	3.01	2.06
8:0:2			2.43	2.00
6:4:0			2.77	1.76
6:0:4			3.48	2.98
4:0:6	26.1	14	5.05	4.74
10:0:0			1.63	2.82
5:5:0			3.18	2.36
5:4:1			3.50	2.79
5:3:2			4.00	3.38
5:2:3			5.13	4.53

C: cement; IP: waste ceramic insulator powder; SP: blast-furnace slag powder; T: temperature (°C); IT: immersion time (days); RL: reaction layer (mm); ND: neutralization depth (mm). Water/(C+IP+SP) = 0.5. RL is the arithmetic mean of five measurements. ND is the arithmetic mean of values calculated as half of the value by deducting the diameter of the pink colored region of the specimen from the diameter of the specimen before immersion.

Table 4 Reaction layer and neutralization depth for mortar in acid resistance tests

Specimen proportion (C:IA:IP:SA:SP)	T (°C)	IT (d)	RL (mm)	ND (mm)
10:20:0:0:0	18.8	29	0.75	2.72
10:16:4:0:0			0.75	2.97
5:20:0:0:5			3.66	2.44
5:16:4:0:5			3.77	2.60
5:14:6:0:5	20.0	33	3.88	2.49
4:20:0:0:6	18.8	29	3.88	2.52
4:16:4:0:6			3.57	2.18
4:14:6:0:6	20.0	33	3.28	2.38
4:0:0:20:6	20.0	32	2.97	1.36
4:0:4:16:6	20.2	31	2.90	1.35
4:8:4:8:6	19.5	32	2.57	1.86
10:20:0:0:0	19.5	57	0.80	5.08
10:16:4:0:0			1.02	6.01
5:20:0:0:5			3.56	3.80
5:16:4:0:5			5.19	4.28
5:14:6:0:5	20.0	63	5.20	4.49
4:20:0:0:6	19.5	57	4.98	4.15
4:16:4:0:6			5.60	4.03
4:14:6:0:6	20.0	63	6.01	4.28
4:0:0:20:6	19.9	63	3.37	2.35
4:0:4:16:6	19.7	59	3.30	2.42
4:8:4:8:6	19.0	63	4.57	3.53
10:20:0:0:0	19.4	86	0.48	8.77
10:16:4:0:0			0.99	9.23
5:20:0:0:5			4.78	6.67
5:16:4:0:5			5.87	5.82
5:14:6:0:5	19.5	94	6.12	6.12
4:20:0:0:6	19.4	86	6.96	5.91
4:16:4:0:6			7.31	5.59
4:14:6:0:6	19.5	94	8.26	6.23
4:0:0:20:6	19.8	96	4.77	3.25
4:0:4:16:6	19.3	87	4.70	3.28
4:8:4:8:6	19.0	84	5.46	4.83
10:20:0:0:0	19.5	115	0.49	12.64
10:16:4:0:0			0.93	13.66
5:20:0:0:5			2.40	9.43
5:16:4:0:5			5.91	7.85
5:14:6:0:5	19.3	115	6.96	7.97
4:20:0:0:6	19.5	115	5.03	8.31
4:16:4:0:6			10.15	7.89
4:14:6:0:6	19.3	115	9.57	8.00
4:0:0:20:6	19.8	112	6.07	4.01
4:0:4:16:6	19.2	115	6.84	4.30
4:8:4:8:6	19.0	112	5.09	6.43

C: cement; IA: waste ceramic insulator aggregate; IP: waste ceramic insulator powder; SA: blast-furnace slag aggregate; SP: blast-furnace slag powder; T: temperature (°C); IT: immersion time (days); RL: reaction layer (mm); ND: neutralization depth (mm). Water/(C+SP) = 0.5 and (IA+IP+SA)/(C+SP) = 2.0.

To determine effective factors for the reaction layer formation, multiple regression analyses were performed with SPSS statistics 22. The dependent variable was the width of the reaction layer. The independent variables were the C part, SP part, IP part, temperature, and immersion time for cement pastes. Regression results of the reaction layer for cement pastes are shown in Table 5. The SP part was removed from the analysis because the ratio had significant relationship with the C and IP parts. The width of the reaction layer increases with variables such as the IP part, temperature, and immersion time, and decreases as the C part increases. The largest standardized coefficient was the immersion time, followed in order by the temperature, IP part, and C part. The results show that the immersion time, temperature, and compounding ratio have an influence on the reaction layer formation. No significant regression result was obtained for mortar because the types and ranges of its independent variables were few.

Table 5 Regression result of reaction layer for cement paste

Variable	B (SE)	β	<i>t</i>	<i>p</i>
(Constant)	-5.104 (1.887)		-2.70	0.015
Cement ratio	-1.891 (0.938)	-0.256	-2.02	0.059
Insulator powder ratio	3.453 (1.024)	0.412	3.37	0.003
Temperature (°C)	0.290 (0.071)	0.547	4.08	0.001
Immersion time (days)	0.087 (0.017)	0.690	5.17	0.000

B(SE): unstandardized coefficients (standard error); β : standardized coefficients; *t*: t-value; *p*: significant. R square = 0.771; adjusted R square = 0.720; standard error of the estimate = 0.787.

3.3 Expansion characteristics of reaction layer

Some reaction layers in the present research were very weak and broke easily. Several studies reported that a specimen expanded when blast-furnace slag was added to concrete [16], [29]. In this study, some specimens had large differences between the reaction layer and the neutralization depth. This is probably because the neutralized parts of the cement paste and mortar expand. Here, the ratio of the reaction layer to the neutralization depth (RL/ND) is considered as an expansion index of the mixture materials.

For cement paste, the relationships between the SP part and the RL/ND, and between the IP part and the RL/ND are shown in Fig. 3. The relationship for the SP part was significantly positive. The results show that cement paste may have expanded with the increasing SP. In contrast, the relationship for the IP

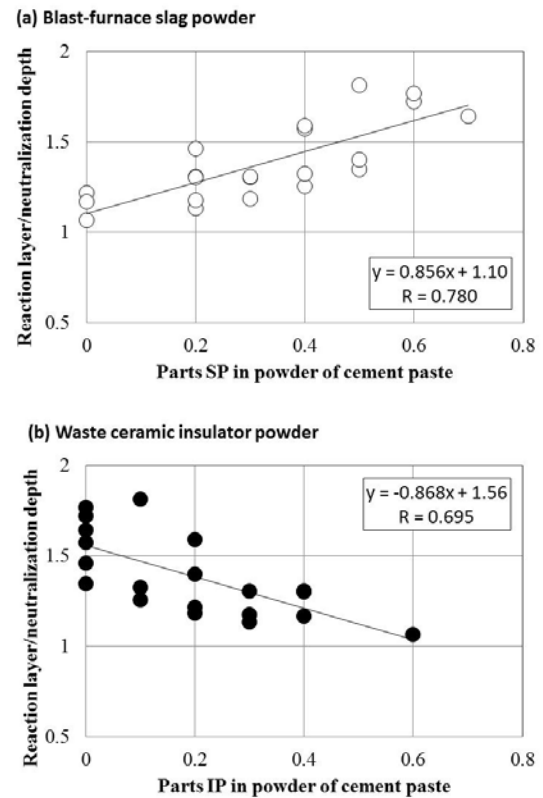


Fig. 3. Relationships between materials in powder of cement paste and ratio of reaction layer to neutralization depth

part was significantly negative. This probably indicates that the waste ceramic insulator can control expansion of the reaction layer for cement paste.

For mortar, the relationships between each part of materials and the RL/ND are shown in Fig. 4. The RL/ND ratios tend to be higher as the SA and SP parts increase, and then the ratio tends to be lower with the increasing IA. The relationship between IP and RL/ND was observed with no tendency because most of the mortars with IP = 0 contained IA. If the data including IA with IP = 0 was removed, the median value of the RL/ND equals to 1.49. These results indicate that the waste ceramic insulator can also control expansion of the reaction layer for mortar.

Adding the waste ceramic insulator properly as an aggregate and/or a powder admixture in cement paste and mortar can contribute to extending the life of concrete structures.

3.4 Elemental composition of reaction layer of cement paste

The element composition of the reaction layers for eight kind of cement pastes (C:IP:SP=10:0:0, 8:2:0, 8:0:2, 5:3:2, 5:2:3, 5:1:4, 5:0:5 and 4:6:0)

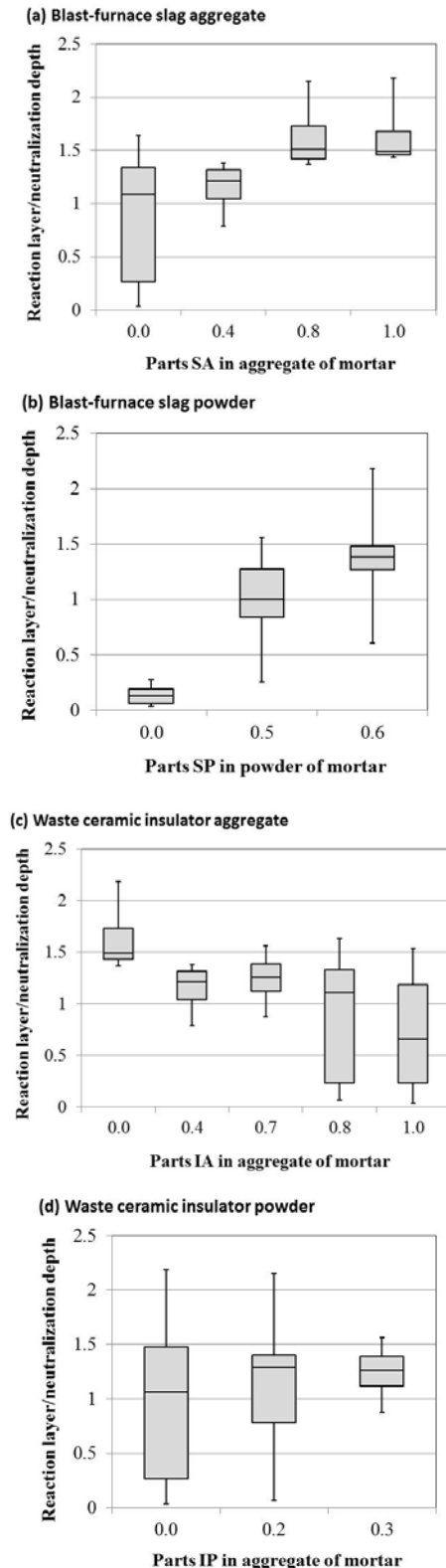


Fig. 4. Relationships between materials in mortar and ratio of reaction layer to neutralization depth. The internal lines across the boxes identify the median values, the ends of the boxes are the 25th percentile and the 75th percentile values, and the whisker bars are the minimum and maximum values.

were measured. The average weight percentages of the elements from the outer side of the layer were 56.0% of O, 22.5% of Si, 10.2% of Ca, 8.0% of S, and 2.6% of Al. The sum of the above five elements was over 97%. The average weight percentages of the elements from the inner side of the layer were 54.8% of O, 14.4% of Si, 14.0% of Ca, 9.7% of S, 3.5% of Al, and 3.2% of Fe. The weight percentages of Si, S and Ca were high. It seems probable that calcium silicate hydrate ($3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$) and calcium hydroxide ($\text{Ca}(\text{OH})_2$) are generated by the hydration reaction of cement, and gypsum dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is generated by the reaction between sulfuric acid and calcium hydroxide. The detected aluminum may be due to ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) generated by the reaction between tri-calcium aluminate acid ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$) and gypsum in cement [30].

4. CONCLUSION

In this study, cement paste and mortar were made from cement, blast-furnace slag, and waste ceramic insulator. The proportions of these materials varied for each specimen. The compressive strength and the acid resistance were investigated. The results are summarized as follows.

(1) The compressive strength of 18 cement pastes was tested. The average compressive strength except two C:IP:SP ratios was more than 24 N/mm^2 , which is the compressive strength standard of sulfuric acid-proof mortar used for a corrosion-protective covering layer. The tests show that the waste ceramic insulator powder can be added by approximately 0.4 parts by weight in the powder materials of the cement paste containing blast-furnace slag.

(2) The compressive strength of 15 mortars was tested. The average compressive strength for all the ratios was more than 24 N/mm^2 . The tests show that the waste ceramic insulator aggregate can be added by two-thirds by weight in the materials of the mortar.

(3) Many specimens with different ratios of cement paste and mortar were immersed in 5% sulfuric acid. A reaction layer formed on the specimen surface for each of all the specimens. The width of the reaction layer was from 1.63 to 8.62 mm for the cement pastes and from 0.48 to 10.15 mm for the mortars. The multiple regression analyses show that the immersion time,

temperature, and compounding ratio of the materials were the influence factors on the reaction layer formation.

(4) The expansion characteristics of the reaction layer were discussed using the ratio of the reaction layer to the neutralization depth (RL/ND). The relationships between the blast-furnace slag ratio and the RL/ND for the specimen were significantly positive. However, those between the waste ceramic insulator ratio and the RL/ND were negative. The results show that adding the waste ceramic insulator to cement paste and mortar can control the reaction layer formation.

5. ACKNOWLEDGEMENTS

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