Sulfuric Acid Resistance of Concrete with Blast Furnace Slag Fine Aggregate

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Abstract: The deterioration of concrete by sulfuric acid attack in sewage environments has become a serious problem for many existing sewage structures. In this study, the properties of concrete using the blast furnace slag have been examined. It was shown that by using the blast furnace slag fine aggregate and blast furnace slag fine powder, it is possible to enhance the resistance of mortar and concrete to sulfuric acid. The resistance to sulfuric acid of mortar and concrete can be improved by using a blast-furnace slag fine aggregate in the total amount of fine aggregate. When mortar or concrete reacts to sulfuric acid, dihydrated gypsum film is formed around the particulate of the fine aggregate. This dihydrated gypsum film could retard the penetration of sulfuric acid, thus, improving the resistance to sulfuric acid. Furthermore, it has been proved that the relationship between the erosion depth by sulfuric acid attack and the product of immersion period and concentration of sulfuric acid can be expressed linearly. However, this relationship is dependent on the type of materials of concrete.

Key words: Sulfuric acid attack, blast furnace slag sand, ground granulated blast furnace slag, gypsum, sewerage.

1. Introduction

Concrete has been used as the main material for constructing civil infrastructure systems such as sewage facilities. In facilities such as these, bacteria react with hydrogen sulfate, producing sulfuric acid which causes the concrete to deteriorate rapidly. There are many methods for protecting concrete from this effect, such as oxygen injection, which prevents the production of hydrogen sulfate, or the application of a product to the surface of the concrete which prevents direct contact with the sulfuric acid [1]. Although these methods are effective, they are mainly used to protect areas of the structure that are most prone to deterioration.

The most efficient way to extend the integrity of the concrete is to develop a concrete with an inherent resistance to sulfuric acid deterioration. This will

protect the whole structure, not only the areas prone to deterioration. A sulfuric acid resistant concrete has been developed using blast furnace slag as an additional material.

Blast furnace slag is a by-product generated during the production of iron. It has been classified as an amorphous material when used as a binder material and called granulated blast furnace slag and classified as a crystalline material when used as a material for road paving, called gradually-cooled blast furnace slag. In Japan, the volume of granulated blast furnace slag produced is over 20 million t per year and 90% of it is used as a material for cement and concrete production [2]. Concrete containing blast furnace slag is well known for improving some properties of concrete such as the watertightness, chemical resistance and resistance to chloride ion permeation [3]. It also improves the resistance to sulfuric acid deterioration. However, when blast furnace slag is added to ordinary cement, such as Portland cement, the resistance to

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sulfuric acid of concrete exhibits the same tendency as ordinary concrete: higher compressive strengths yield a lower resistance to sulfuric acid. A greater amount of cement hydrate in concrete which has a smaller water to binder ratio causes a greater amount of product from the reaction of sulfuric acid and cement hydrate [4-6]. If ground granulated blast furnace slag is added to Portland cement, it can improve the resistance to sulfuric acid.

It is known that the sulfuric acid resistance can be improved when Portland cement is combined with ground granulated blast furnace slag. On the other hand, when the ground granulated blast furnace slag is used as a fine aggregate, it forms a dihydrated gypsum film around its own particle. These gypsum films could impede further penetration of the sulfuric acid into the concrete. It has been proved in this study that a low water to binder ratio produces a higher amount of the dihydrated hardener which gives the concrete a higher resistance to sulfuric acid. In this study, the use of granulated blast furnace slag as a fine aggregate in high strength concrete improved the resistance to sulfuric acid.

2. Sulfuric Acid Immersion Test

2.1 Materials and Mixed Proportions

Ordinary Portland cement (density: 3.15 g/cm^3 , Blaine size: $3,300 \text{ cm}^2/\text{g}$) and ground granulated blast

furnace slag (density: 2.89 g/cm^3 , Blaine size: $4,150 \text{ cm}^2/\text{g}$) are used. River sand, granulated blast furnace slag (called blast furnace slag sand) and crushed limestone sand are used as fine aggregates. Crushed stone is used as a coarse aggregate. The properties of the aggregates are shown in Table 1. The polycarboxylate type of high range water reducing admixture is used as an additional admixture. Mixed proportions of cement paste, mortar and concrete are shown in Tables 2-5. The dosage of admixture is determined by a decided flow for mortar and a slump flow for concrete. The decided flow values for mortar are about 250 mm for mortar specimens with water to binder ratios of 25% and 30%, and about 180 mm for mortar specimens with a water to binder ratio of 60%. The decided slump flow value for concrete is about 650 mm for concrete specimens with a water to binder ratio of 25%, while the decided slump value for concrete is 5 cm for specimens with a water to binder ratio of 60%.

2.2 Experimental Method

Particle sizes (0.3 mm to 5.0 mm) of fine aggregates were used. The samples were prepared in an absolutely dry state. A 120 g sample of each aggregate and 200 mL of 5% by mass concentration of sulfuric acid were used for each test. After the samples were immersed in sulfuric acid for 14 days, each

Table 1 Properties of aggregate.

Type of aggregate		Saturated density (g/cm^3)	Water absorption rate (%)	Fineness modulus
Fine aggregate	River sand	2.61	1.97	2.96
	Blast furnace slag sand	2.77	0.72	2.15
	Crushed limestone sand	2.68	0.70	2.79
Coarse aggregate	Crushed sandstone	2.75	0.54	6.73

Table 2 Mix proportion of cement paste.

Water to binder ratio (%)	Cement to binder ratio (%)
30.0	40.0
	100.0
60.0	40.0
	100.0

Binder: ordinary Portland cement and ground granulated blast furnace slag powder.

Table 3 Mix proportion of mortar experimenting on reaction of sulfuric acid to fine aggregate.

W/B (%)	C/B (%)	Air (%)	Unit content (kg/m ³)						HRWRA ^{*5} (kg/m ³)
			W	OPC	BF ^{*1}	RS ^{*2}	BFS ^{*3}	LS ^{*4}	
30.0	100.0		213	710	0	1,414	0	0	12.78
						0	1,500	1,451	
31.6	40.0	2.0		269	404	1,414	0		5.38
						0	1,500		
60.0	100.0		287	478	0	1,414	0	0	0.00
						0	1,500		
63.2	40.0			181	272	1,414	0		
						0	1,500		

^{*1} BF: ground granulated blast furnace slag powder; ^{*2} RS: river sand; ^{*3} BFS: blast furnace slag sand; ^{*4} LS: limestone sand; ^{*5} HRWRA: high-range water reducing admixture.

Table 4 Mix proportion of mortar on experimenting of deterioration rate of sulfuric acid.

W/B (%)	C/B (%)	Air (%)	Unit content (kg/m ³)					HRWRA ^{*4} (kg/m ³)
			W	OPC	BF ^{*1}	RS ^{*2}	BFS ^{*3}	
25.0	100.0	2.0	220	880	0	1,250	0	13.20
	40.0			352	528	0	1,271	
60.0	100.0			367	0	1,673	0	0.00
	40.0			147	220	0	1,740	

^{*1} BF: ground granulated blast furnace slag powder; ^{*2} RS: river sand; ^{*3} BFS: blast furnace slag sand; ^{*4} HRWRA: high-range water reducing admixture.

Table 5 Mix proportion of concrete.

G_{max} (mm)	W/B (%)	C/B (%)	Air (%)	s/a (%)	Unit content (kg/m ³)						HRWRA ^{*5} (kg/m ³)
					W	OPC	BF ^{*1}	RS ^{*2}	BFS ^{*3}	CS ^{*4}	
20	25.0	100.0	2.0	45.0	175	700	0	684	0	881	0.65
		40.0				280	420	0	711	865	0.55
	60.0	100.0				292	0	834	0	1,074	1.17
		40.0				117	175	0	869	1,066	

^{*1} BF: ground granulated blast furnace slag powder; ^{*2} RS: river sand; ^{*3} BFS: blast furnace slag sand; ^{*4} CS: crushed stone; ^{*5} HRWRA: high-range water reducing admixture.

sample was removed from the container, washed of sulfuric acid by running water, then dried out in a 105 ± 5 °C temperature controlled oven for 12 h. The dried samples were sieved, weighed and measured, the sample material remaining at the 0.3 mm sieve were weighed as well.

The specimen was a cylinder specimen with a 50 mm diameter and 100 mm height for cement paste and mortar. The concrete specimen was a cylinder specimen with a 100 mm diameter and 200 mm height. The specimens were cured up to age 7 days in water after remolding.

The concentrations 1%, 3%, 5% and 10% by mass of

sulfuric acid were prepared. The volume of sulfuric acid for each test was calculated by a 1:2 mass ratio of sulfuric acid to specimen. The total amount of sulfuric acid was replaced every 7 days. The mass of the specimens were measured every 7 days as well. At a predetermined date, the specimens were cut and sprayed with a phenolphthalein solution on the cutting face and the diameter of the coloration area was measured.

3. Results of Sulfuric Acid Immersion Test

3.1 Deterioration of Sulfuric Acid to Cement Paste

The cycle of deterioration of sulfuric acid to cement

paste is shown in Fig. 1. Fig. 1a shows the condition of the cutting surface of the specimen after immersion in sulfuric acid. Fig. 1b shows a state where dihydrated gypsum film is formed by the reaction of calcium and sulfuric acid. Fig. 1c shows a state where ettringite is generated at the contact surface of cement and dihydrated gypsum, when tricalcium aluminate in cement reacts with dihydrated gypsum. Fig. 1d shows a state where formed ettringite changes to become a paste-type gypsum when it comes into contact with penetrated sulfuric acid, making a lower pH in the cement paste. Fig. 1e shows a state where ettringite reacts with penetrated sulfuric acid. The reaction increases the paste-type of dihydrated gypsum and it peels away from the cement surface. Fig. 1f shows the state where the remaining ettringite makes direct contact with sulfuric acid, becoming a paste-type dihydrated gypsum, finally peeling away from the cement surface. This entire cycle is then continually repeated [1, 7].

The result of a mass change in the cement paste specimen when 5% concentration of sulfuric acid was used in the immersion tests is shown in Fig. 2. The symbols ○ and ● in the figure represent the results

of ordinary Portland cement specimens with water to cement ratios of 30% and 60%, respectively. Ordinary Portland cement with a 30% water to cement ratio shall be referred to as OPC30 and ordinary Portland cement with a 60% water to cement ratio shall be referred to as OPC60. The symbols □ and ■ in the figure represent the result of cement specimens containing blast furnace slag with water to binder ratios of 30% and 60%, respectively. Cement containing blast furnace slag with a 30% water to binder ratio shall be referred to as BB30 and cement containing blast furnace slag with a 60% water to binder ratio shall be referred to as BB60. The ratio of cement and blast furnace slag is 4:6 by mass.

The mass loss tendency of OPC30 is linear. It reaches -72% when the immersion time is 56 days. While OPC60 has two cycles of gaining a little mass at the beginning and losing mass at 56 days. BB30 has a cycle of gain and loss of mass during the 56-day test, while BB60 gains mass and has no cycle of losing mass.

The results indicate that smaller water to cement ratios yield a higher strength cement product. However, there is also a higher rate of loss of mass. When blast furnace slag is used, the mass loss of the

Fig. 1 Cycle of deterioration of cement paste due to sulfuric acid immersion test.

Fig. 2 Result of sulfuric acid immersion test.

specimen is lower than the specimen that contains ordinary Portland cement. The research of Kurashige et al. [8] and others explains the lower resistance to sulfuric acid of concrete as a result of the greater presence of pores in the microstructure of concrete that has a high water to cement ratio. These pores could suppress the stress caused by dihydrated gypsum which is produced by the reaction of sulfuric acid and concrete.

Fig. 3 shows the cutting surface of a specimen after the immersion test has passed 56 days. The diameter of the specimen before the immersion test is 50 mm. There is no gypsum film remaining at the surface of the OPC30 specimen which shows a high deterioration of the specimen caused by sulfuric acid. OPC60 shows an amount of gypsum remaining around the specimen, but the diameter of the coloration area has decreased. BB30 and BB60 show an amount of gypsum remaining, creating a larger diameter of the specimen compared with its diameter before the immersion test. The coloration by phenolphthalein solution indicates the area that does not react with sulfuric acid. The BB specimens have a greater diameter than the OPC specimens.

The ordinary Portland cement mixture confirms that a smaller water to cement ratio yields a smaller diameter of coloration by phenolphthalein solution.

Fig. 3 Cutting surface of cement paste specimens after 56 days of sulfuric acid immersion test.

However, the mixture containing blast furnace slag demonstrates an opposite tendency. The water to cement ratio has less influence on the loss of mass and carbonation of the specimen. When the gypsum is formed, it prevents the elution of decomposition of the product as well as the penetration of sulfuric acid into the specimen [9], thus, improving the sulfuric acid resistance of the specimen.

3.2 The Reaction of Fine Aggregate to Sulfuric Acid

Fig. 4 shows mass loss of fine aggregate particle sizes (0.3 mm to 5.0 mm) after the sulfuric acid immersion test. The symbols $\square$, $\circ$ and $\triangle$ in the figure represent the result of river sand, blast furnace slag sand and crushed limestone sand, respectively. The river sand shows a mass loss of 20% during the 56 day immersion test. It is the lowest reactivity with sulfuric acid compared to the other fine aggregate. On the other hand, when the crushed limestone sand was immersed into sulfuric acid, it generated carbon dioxide gas, which indicates a strong reaction. The mass loss is over 50% after the completion of the 56 days immersion test. The blast furnace slag shows a low number of mass loss. In fact, the mass actually increased after 14 days of the immersion period. This is due to a reaction of the blast furnace slag with sulfuric acid which produces dihydrated gypsum on the surface of the sample. It has been observed that,

Fig. 4 Mass loss of fine aggregate due to the sulfuric acid immersion test.

due to high amounts of calcium compounds in both blast furnace slag sand and crushed limestone sand, both aggregates have strong reactions with sulfuric acid, though the products are different.

3.3 Deterioration of Mortar to Sulfuric Acid

Fig. 5 shows the result of the sulfuric acid immersion test of mortar containing different types of fine aggregate and a 30% water to cement ratio. The ordinary Portland cement has been used as the binder material. The symbols $\square$, $\circ$ and $\triangle$ in the figure represent the results of river sand, blast furnace slag sand and crushed limestone sand, respectively. Among these three aggregates, river sand has the highest mass loss, followed by limestone and, finally, with the lowest loss of mass, blast furnace slag. As a result, the lowest mass loss of a fine aggregate when it reacts with sulfuric acid is river sand, which has the highest mass loss of a specimen when it is used in mortar. Aggregates with a higher reactivity with blast furnace slag sand, such as crushed limestone stone and blast furnace slag sand, have a higher resistance to sulfates when they are used in mortar, especially blast furnace slag which, when used as a fine aggregate, shows insignificant data of mass loss during the 56 days of sulfuric acid immersion test.

Fig. 6 shows the comparison of the cutting surface

of cement paste and mortar specimens with the same water to binder ratio. River sand was used as a fine aggregate in the mortar specimen. The diameter of the specimen before the immersion test was 50 mm. Fig. 6a shows the test results of the cement paste specimens, while the results of the mortar specimens are shown in Fig. 6b. The mortar specimens show that there is no gypsum paste remaining on the surface of specimens. However, there is dihydrated gypsum film remaining on the same mixture of the cement paste specimen. In addition, the smaller water to binder ratio shows a greater effect of erosion by sulfuric acid.

Fig. 7 shows the comparison of the cutting surface of the cement paste specimens and the mortar specimens

Fig. 5 Result of mass loss of mortar specimens.

Fig. 6 Result of sulfuric acid immersion test of: (a) cement paste; (b) mortar with river sand.

Fig. 7 Result of sulfuric acid immersion test of: (a) cement paste; (b) mortar with blast furnace slag sand.

with the same water to binder ratio. Blast furnace slag sand was used as a fine aggregate in the mortar specimens. The sulfuric acid immersion test results show that even the OPC30 cement paste specimen lost all trace of the dihydrated gypsum. However, the dihydrated gypsum was formed in the mortar specimen. The depth of carbonation becomes more shallow when blast furnace slag sand is used as a fine aggregate. Furthermore, the cement paste mixture, which has dihydrated gypsum, becomes resistant to sulfuric acid when blast furnace slag is used as a fine aggregate.

Fig. 8 shows the formation of dihydrated gypsum film on mortar which has river sand as a fine aggregate. As there is a cavity between the bond of the particle of river sand and the dihydrated gypsum film, the gypsum is porous. Conversely, Fig. 9 shows a highly dense dihydrated gypsum film at the surface of mortar which has blast furnace slag sand as a fine aggregate. It has been observed that, even when the ground granulated blast furnace slag powder has been combined with the binder, the density of the dihydrated gypsum film which is produced by the reaction of sulfuric acid differs greatly. The component of blast furnace slag sand is substantially the same as the component of cement and the reaction rate to sulfuric acid is nearly the same as well. It is considered that the dihydrated gypsum film produced

by the reaction of sulfuric acid and mortar which has blast furnace slag sand is homogeneous. On the other hand, when the river sand is used, the dihydrated gypsum film is heterogeneous at the boundary of the sand particle. Due to the cavity around the particle and the heterogeneous formation, the dihydrated gypsum is not strong enough to prevent the penetration of sulfuric acid. Thus, the mortar becomes more fragile than cement paste when under sulfuric acid attack.

3.4 Deterioration of Concrete to Sulfuric Acid

The results of the concentration 5% by mass of sulfuric acid immersion test of concrete are shown in Fig. 10. The symbols $\square$ and $\circ$ in the figure represent the results of normal concrete using river sand and blast furnace slag sand as a fine aggregate, respectively. Crushed sandstone is used as a coarse aggregate and the water to binder ratio of all mixtures was 25%. Ordinary Portland cement was used as a binder for the normal concrete mixture and ground

Fig. 8 Surface of mortar with river sand.

Fig. 9 Surface of mortar with blast furnace slag sand.

Fig. 10 Sulfuric acid resistance of mortar containing river sand and blast furnace slag.

granulated blast furnace slag powder was used as a binder material in concrete using the blast furnace slag sand mixture. The combined amount of blast furnace slag in the binder is 60%. The mass loss of normal concrete reached 30%, while the concrete with blast furnace slag powder and sand has a relatively small change in mass.

Fig. 11 shows the cutting surface of concrete containing blast furnace slag after 56 days of the sulfuric acid immersion test. The size of the specimen was 100 mm in diameter before the test. It can be seen that the dihydrated gypsum was formed and that it prevented the penetration of sulfuric acid into the concrete. The sulfuric acid resistance has been improved.

3.5 Prediction of Erosion Depth of Concrete with Sulfuric Acid Attack

Fig. 12 shows the relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and the immersion period of normal mortar—the mortar using ordinary Portland cement as a binding material and river sand as a fine aggregate. The symbols ■, ▲ and ● in the figure represent the immersion results of normal mortars with a 25% water-binder ratio in 1%, 3% and 5% sulfuric acid at a concentration by mass%, respectively, while □, △ and ○ represent the results of normal mortar with a

60% water to binder ratio in the previously mentioned sulfuric acid concentrations, respectively. The relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and the immersion period has been established as linear. The normal mortar with a 25% water to binder ratio shows a deeper slope when compared to normal mortar with a 60% water to binder ratio. The corrosion depth of normal mortar with a 25% water to binder ratio is 13.0 mm/(year·%), while the corrosion depth of normal mortar with a 60% water to binder ratio is 9.4 mm/(year·%). The lower strength mortar experiences greater corrosion depth due to sulfuric acid than the higher strength mortar.

On the other hand, the results of mortar containing blast furnace slag sand and slag mortar are shown in Fig. 13. The ground granulated blast furnace slag was used in a proportion of 4:6 by mass ratio of ordinary

Fig. 11 Carbonation of concrete containing blast furnace slag after sulfuric acid immersion test.

Fig. 12 Relationship between the concentration of sulfuric acid, soaking period and erosion depth of normal mortar.

Portland cement and ground granulated blast furnace slag. The bolt and blank drawing represents the results of immersion of slag mortar with 25% and 60% water to binder ratios in 1%, 3% and 5% sulfuric acid at a concentration by mass%, respectively. The relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and immersion period has been also established as linear. The corrosion depth of slag mortar with a 25% water to binder ratio is 2.6 mm/(year·%), while the corrosion depth of slag mortar with a 60% water to binder ratio is 3.0 mm/(year·%). The use of blast furnace slag sand in mortar has less of an effect on corrosion depth of sulfuric acid attack than the water to binder ratio does. In addition, when the same water to binder ratio of normal and slag mortar has been compared, the slag mortar shows a smaller depth at the same concentration of sulfuric acid. The depth is 13 mm/(year·%) of normal mortar with a 25% water to binder ratio, while it is 2.6 mm/(year·%) for slag mortar with the same water to binder ratio. The corrosion depth has become five times smaller when the blast furnace slag is used.

Fig. 14 shows the relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and the immersion period of normal concrete, concrete using ordinary Portland cement as a binder and river sand as fine aggregate. The symbols ■, ● and ▼ in the figure represent the immersion results of normal mortars with a 25% water to binder ratio in 1%, 5% and 10% sulfuric acid at a concentration by mass%, respectively, while □, ○ and ▽ represent the results of normal concrete with a 60% water to binder ratio for the previously mentioned sulfuric concentrations, respectively. As with the mortars, the relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and the immersion period has been established as linear. The corrosion depth of normal concrete with a 25% water to binder ratio is 10.1 mm/(year·%), while the corrosion depth of normal

mortar with a 60% water to binder ratio is 4.5 mm/(year·%). It can be seen that, as the strength of normal concrete increases, the resistance to sulfuric acid attack increases. Furthermore, higher strengths actually reduce the resistance to sulfuric acid in concrete while they increase the resistance in mortar.

The results of concrete, containing blast furnace slag sand and slag concrete, are shown in Fig. 15. The ground granulated blast furnace slag was used in a proportion of 4:6 by mass ratio of ordinary Portland cement and ground granulated blast furnace slag. The bolt and blank drawing represents the results of the immersion of slag concrete with 25% and 60% water to binder ratios in 1%, 5% and 10% sulfuric acid at a

Fig. 13 Relationship between the concentration of sulfuric acid, soaking period and erosion depth of slag mortar.

Fig. 14 Relationship between the concentration of sulfuric acid, soaking period and erosion depth of normal concrete.

Fig. 15 Relationship between the concentration of sulfuric acid, soaking period and erosion depth of slag concrete.

concentration by mass%, respectively. The relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and immersion period has been established as linear. The corrosion depth of slag concrete with a 25% water to binder ratio is 2.6 mm/(year·%), while the corrosion depth of slag concrete with a 60% water to binder ratio is 4.6 mm/(year·%). The use of blast furnace slag sand in concrete has less of an effect on the corrosion depth of sulfuric acid attack than the water to binder. In addition, when the same water to binder ratio of normal and slag concrete has been compared, the slag concrete shows a smaller depth at the same concentration of sulfuric acid. The depth is 10.1 mm/(year·%) for normal concrete with a 25% water to binder ratio, while it is 2.6 mm/(year·%) for slag concrete at the same water-binder ratio. The corrosion depth has become four times smaller when the blast furnace slag is used.

4. Conclusions

The following conclusions can be drawn from this study:

- (1) When river sand is used as a fine aggregate in mortar and concrete, higher strengths produce a low resistance to sulfuric acid;
- (2) The resistance to sulfuric acid of blast furnace

slag sand and crushed limestone sand are lower than that of river sand. However, the sulfuric acid resistance of mortar with blast furnace slag sand and crushed limestone sand is higher than that of mortar with river sand;

(3) When blast furnace slag sand is used, the dihydrated gypsum film formed by the reaction of sulfuric acid and blast furnace slag sand is more dense and can prevent the penetration of sulfuric acid to the interior of the cement specimen. In addition, a lower water to binder ratio produces higher strength and sulfuric acid resistance;

(4) The relationship between the product of the concentration of sulfuric acid and sulfuric acid erosion depth, and immersion period has been established as linear;

(5) The stiffness of the dihydrated gypsum, as well as the resistance to sulfuric acid of mortar and concrete in a running water environment, should be investigated. Finally, the microstructures of mortar and concrete with blast furnace slag are more dense than the microstructures of ordinary mortar and concrete. The use of blast furnace slag sand to improve the resistance to chloride ion permeability should be investigated.

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