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# Materials Research Express



## PAPER

# Investigation of ferronickel slag powder for marine applications by using MIP method

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**Keywords:** permeability test, pore structure, marine environment, impermeability, ferro-nickel slag powder

## Abstract

The main aim of this work is to study the performance of the ferro-nickel slag in the powder form and to analyze the permeability performance using MIP method. The test is carried out under two different conditions. It is performed in clean water and a marine environment. Under these conditions different admixture conditions were examined. The change in impermeability of soil and cements for various admixture conditions were examined and the values were measured. A Mercury Intrusion Porosimeter (MIP) was used to examine the pore structure. Permeability values shown increasing trend in both clean water and in a marine environment. The Impermeability values shown increasing trend in marine environment compared with the clean water. During experimentation when Ferronickel slag powder exceeds 20 percentage. The impermeability reduces gradually and it gets decreased. 40 percentages of ferronickel slag powder obtained were seemed to be optimum. Based on the results obtained from Mercury Intrusion Porosimeter (MIP), synergetic integration of ferro nickel slag powder on mixing with the soil-cement creates a micro-aggregate effect and the porosity of soil-cement which in turn increased the resistance for sea water erosions.

## 1. Introduction

More and more attention has been given to the development of ecological and energy-saving engineering materials for use in foundation treatment projects. By mixing a certain amount of ferronickel slag powder into the soil-cement to replace the cement, not only can the amount of cement in the soil-cement be reduced, but more importantly, this process can recycle and reuse nickel waste [1–3]. Additionally, the lower price of ferronickel slag powder can reduce the overall engineering construction cost [4], which is not only in-line with a green development concept in China but is also in-line with current reforms for modern engineering material technology innovation and industrial transformation.

At present, soil-cement has been widely used in engineering construction [5–7], especially to stop water and for seepage prevention, meaning that soil-cement is often used in corrosive site environments [8]. The coastlines in China are long. For coastal construction projects, the soil-cement in a marine environment will be eroded by corrosive substances in the seawater for a long time, this affects the mechanics and the impermeability of the soil-cement. The degradation of strength and impermeability could be relieved by increasing the cement content and incorporating supplementary cementations materials such as slag and fly ash [9, 10]. Therefore, it is a popular research topic to add mineral admixtures into soil-cement to improve its durability and reduce engineering costs. Ma Cong *et al* [11] found that the supplementary cementing materials consisting of sodium silicate and composite promoter performed effectively pozzolanic reactions and improved the mechanical properties of cement stabilized soil. Chairat and Panich [12] found that cement and fly ash geopolymer can be effectively used for stabilized marginal lateritic soil as road material. Goreham *et al* [13] studied the influence of

water on the diffusion and porosity parameters of cement soil materials. Wang *et al* [14] based on the existing researches, the permeability of reactive magnesia (MgO)-carbonated soils is highlighted for its application in engineering. Chen [15] studied the effect of adding ferronickel slag powder on the early strength of cement soil, and the results showed that the early age ferronickel slag powder had weak strengthening effect. Chew *et al* [16] and Ouhadi [17], have studied the mechanism of cemented soil solidification from the micro level by means of micro test method. Heineck *et al* [18] Studied the cement soil affected by alkaline pollutants and obtained the law of strength attenuation. Huang *et al* [18] found that the appropriate reinforcement of polypropylene fibers and cement is an effective way to recycle heavy metal contaminated soil (HMCS) as substitutable fillers in roadbed. Cheng *et al* [19] investigated the primary yielding and yield locus for cement stabilized marine clay. Wang *et al* [20] Investigated the durability and swelling of solidified/stabilized dredged marine soils with class-f fly ash, cement, and lime.

Based on the literatures carried out various works have been carried out to improve the performance of the soil-cement in corrosive environments by adding admixtures. However, there are few literature only reported on the use of ferronickel slag powder to enhance the impermeability of soil-cement, and proper attention has not been paid to the overall strength and impermeability of soil-cement in a marine environment. This paper, therefore, discusses mixing industrial waste ferronickel slag powder into soil-cement and the permeability characteristics of soil-cement once mixed with ferronickel slag powder in a marine environment.

## 2. Permeability test for cement soil

### 2.1. Test material

The water used in the preparation of the soil-cement and curing solution in the test was purified water taken from tap water, then purified using a model WP-RO-10B ultrapure water machine. The soil material used was taken from the foundation pit of a certain subway station. The soil material was the marine sediments slit from the Changle Formation from the Holocene Quaternary Strata, with a water content of 58.5%, a weight of  $1600 \text{ kg m}^{-3}$ , and a porosity ratio of 1.53. Ordinary Portland cement (P.O 42.5) was taken. Due to the weak early activity of ferronickel slag powder, it was necessary to mix part of the granulated blast-furnace slag to stimulate its activity. The blast-furnace ferronickel slag powder and granulated blast-furnace mineral powder used were all from Fujian Yuanxin Environmental Protection Technology Co., Ltd. The mass ratio of ferronickel slag powder to mineral powder was 2:1, and the basis for the mixture's improvement and the chemical composition of mineral additives were referred to in the literature [21].

### 2.2. Test proposal

Initially a bar in the rounded form is taken for examination. Standard procedure was used as per the OEM Standard to prepare the specimen. Dust and Rust present is removed initially and after removing the rust, The specimen is polished and kept in a over for more than 6 h duration for better curing. In this work the steel bars were impregnated with epoxy resin in the ratio 10:1. Once preparation is completed, the cement slurry was dispensed into the wet soil and synergized consistently by means of a grout mixer. On completing this proper curing was carried out for the duration of 48 h finally test molds were removed.

A pressurized permeability device was used to carry out permeability tests on the ferronickel slag powder soil-cement. The pressure device used the constant head method to determine the permeability coefficient of the ferronickel slag powder soil-cement, thereby improving the test efficiency and the accuracy of the results. Three influencing factors were set for the test: the admixture amount of ferronickel slag powder, the environmental conditions of the soil-cement, and the curing period. The quality of the ferronickel slag powder, etc instead of cement quality 0%, 10%, 20%, 30%, and 40% was taken as a variable to study the impact for a clean water environment and a marine environment on the impermeability of the ferronickel slag powder soil-cement at ages of 7d, 28d, 60d, and 90d. Namely, 120 standard soil-cement samples were taken with the water content of soil-cement, water-binder ratio, and total binding material mixing ratio as constants, and the admixture of ferronickel slag powder, curing environment and curing period as variables. The permeability test plan for soil-cement is shown in table 1.

The site conditions of the soil-cement were designed to simulate a marine environment, and the two environments for curing conditions, in clean water and curing conditions in seawater, were used for comparison. Similarly, the formed soil-cement samples were put into the standard curing room with molds for 48 h, and then the metal test molds were removed. Next, the samples without molds were put into the clean-water curing box and the seawater curing box separately to immerse and cure until the desired age.

**Table 1.** Permeability test proposal.

Designation no.	Cement (%)	Water cement	Mixing ratio (%)	Mass Ratio	Environment
A-0	15	0.5	0	0	Fresh water
A-1	15	0.5	10	10	Fresh water
A-2	15	0.5	20	20	Fresh water
A-3	15	0.5	30	30	Fresh water
A-4	15	0.5	40	40	Fresh water
B-0	15	0.5	0	0	Seawater
B-1	15	0.5	10	10	Seawater
B-2	15	0.5	20	20	Seawater
B-3	15	0.5	30	30	Seawater
B-4	15	0.5	40	40	Seawater

Notes: Mixing ratio—a: the ratio of ferronickel slag powder to replace cement mass.

**Table 2.** Permeability coefficient of ferronickel slag powder soil-cement.

Mix proportion	Age	A-0	A-1	A-2	A-3	A-4	B-0	B-1	B-2
Permeability coefficient ( $10^{-8} \text{ cm s}^{-1}$ )	7d	4.83	3.9	2.05	1.75	0.68	5.14	4.01	2.16
	28d	4.47	3.73	1.91	1.59	0.37	5.91	4.21	2.46
	60d	4.24	3.18	1.63	0.88	0.31	7.81	6.47	2.96
	90d	3.83	3.08	1.07	0.51	0.19	9.24	7.71	4.31

Note: The ratioNo. is the same as in table 1.

### 2.3. Test process

The TJSS-25 soil-cement permeability device from Fujian Jiangxia University's Collaborative Innovation Center of Environmental Protection and Energy-saving in High-performance Concrete was used for this test. Before the test, paraffin was used to seal the ferronickel slag powder soil-cement to stop the water. The operational steps of the test were subject to the steps for the permeability test in the *Specification for Mix Proportion Design of Soil-cement* (JGJ/T 23). The permeability coefficient of ferronickel slag powder soil-cement was also calculated based on Darcy's Law and the permeability coefficient measurement requirements in the specification. A water temperature of  $20^\circ\text{C}$  was taken as the standard temperature for the soil-cement permeability test. Therefore, it was necessary to correct the permeability coefficient for this water temperature  $T^\circ\text{C}$ , and obtain the soil-cement permeability coefficient at this standard temperature of  $20^\circ\text{C}$  through correction. The dynamic viscosity coefficient  $\eta_T$  of the water for the test should comply with the relevant regulations in the Standard for Geotechnical Testing Method (GB/T50123–2019).

### 2.4. Permeability test results

The permeability coefficient test calculation for soil-cement samples that were immersed and cured until the desired age in a clean water environment and a marine environment was carried out. After the average value was taken and the requirements were met, through correction, the permeability coefficient of the ferronickel slag powder soil-cement with different mix ratios in a clean water environment and a marine environment could be obtained. The results obtained are shown in table 2.

### 2.5. Analysis of the permeability test results

According to table 2, the changes in the relationship between the permeability coefficient of the soil-cement mixed with ferronickel slag powder and the admixture amount of ferronickel slag powder in the clean water environment and the marine environment were drawn in the paper, as shown in figures 1 and 2. At the same time, a comparative curve diagram of the permeability coefficient of the ferronickel slag powder soil-cement under two immersion environments was drawn, as shown in figure 3.

From the changing of the curves in figures 1 and 2, the overall trend of the soil-cement permeability coefficient at each age can be roughly judged. The permeability coefficients of the soil-cement of each mixing ratio in the two immersion curing environments were compared with the respective baseline group permeability coefficients, and the decreased rate with a fixed base of the soil-cement due to the change of the ferronickel slag powder content could be obtained, as shown in table 3.

The effects of the impermeability of the ferronickel slag powder soil-cement were analyzed and displayed in figures 1–3 and table 3 as follows:

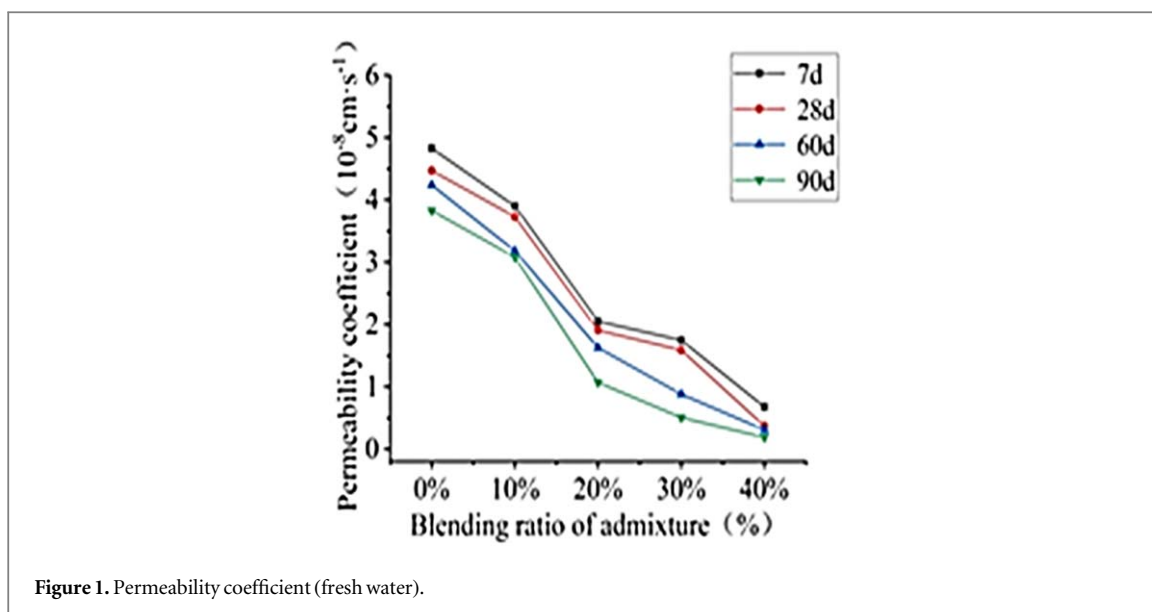


Figure 1. Permeability coefficient (fresh water).

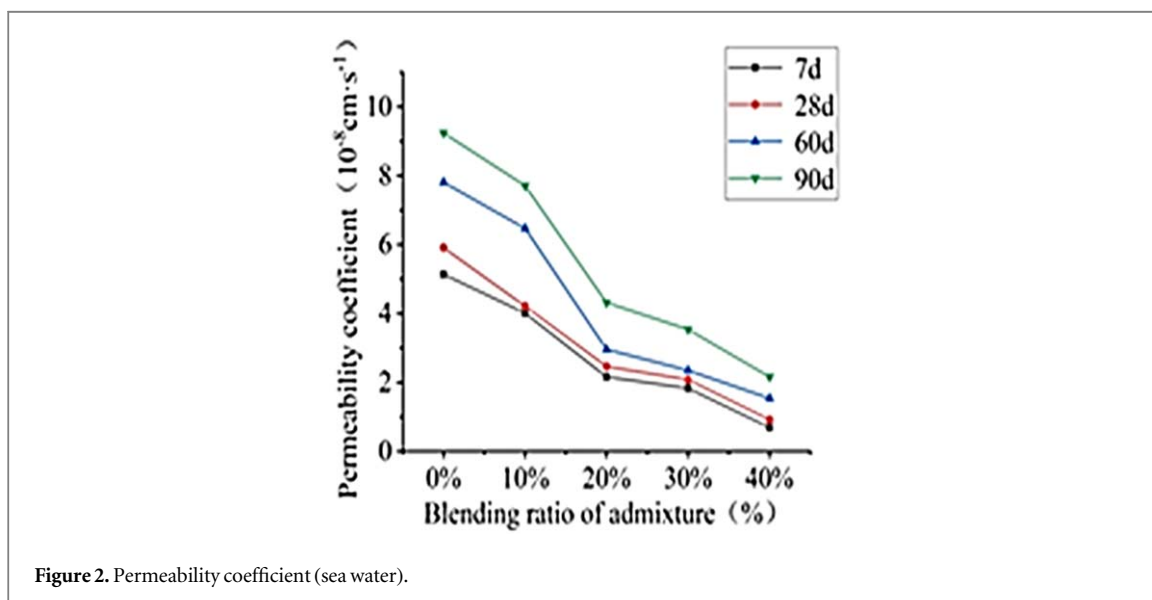


Figure 2. Permeability coefficient (sea water).

- (1) 7d Immersion age: From figures 1–3(a), it can be seen that a downward trend with an increase of ferronickel slag powder content is shown in the permeability coefficient curves of soil-cement in the clean water environment and the marine environment. Additionally, the trend of the curves in the two environments are mostly the same. When the admixture increases to 40%, the two curves basically coincide. From table 3, it can be seen that the permeability coefficient of ferronickel in the clean water environment and the marine environment has decreased to varying degrees compared with the baseline group. It shows that at 7d old, its impermeability improves with the increase of the admixture, and the impact of the environment on the impermeability of soil-cement at an early age is little.
- (2) 28d Immersion age: From figures 1–3(b) and table 3, it can be seen that the permeability coefficient curve of the clean water environment is lower than that of the marine environment, showing that the permeability coefficient of soil-cement in a marine environment is greater than that in a clean water environment. Soil-cement's impermeability can be enhanced by an admixture of ferronickel slag powder to soil-cement, while the impermeability of soil-cement is slightly reduced in the marine environment. The reason is that the solidification of soil-cement continues. Ferronickel slag powder not only begins to play an active role but also plays a role of micro-aggregate effects which makes the soil-cement more compact. Therefore, its impermeability can be improved by an admixture of ferronickel slag powder.
- (3) 60d Immersion age: from figures 1–3(c) and table 3, it can be seen that the increase in the mixing ratio of ferronickel slag powder has a greater effect on the impermeability of soil-cement. However, when the

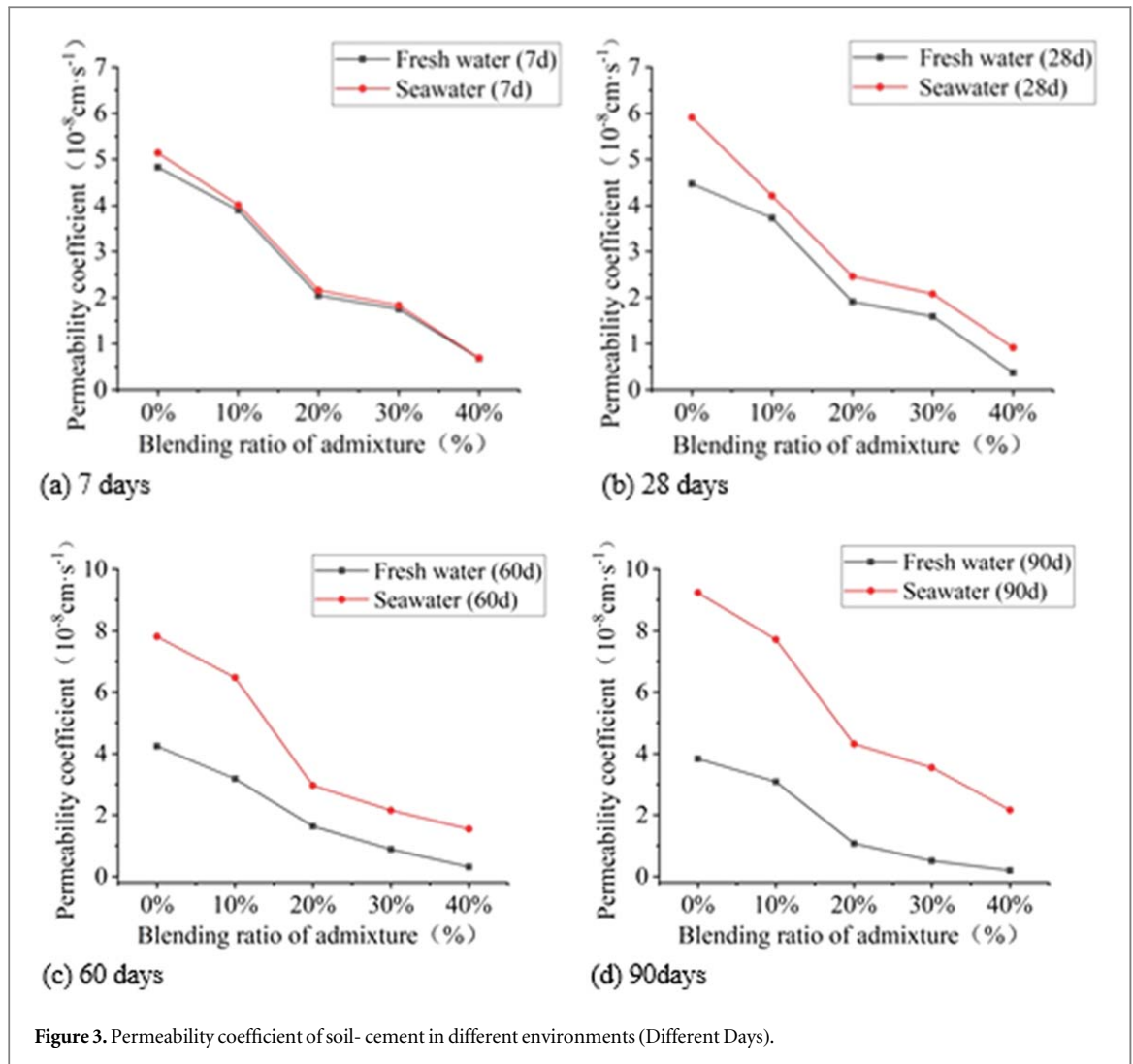


Figure 3. Permeability coefficient of soil- cement in different environments (Different Days).

Table 3. Decrease rate of soil-cement permeability coefficient.

Rate of permeability coefficient change (%)	AGE	A-1 to A-0	A-2 to A-0	A-3 to A-0	A-4 to A-0	B-1 to B-0	B-2 to B-0	B-3 to B-0	B-4 to B-0
	7d	19.25	57.56	63.77	85.92	21.98	57.98	64.40	86.58
	28d	16.55	57.27	64.43	91.72	28.76	58.38	64.81	84.50
	60d	25.00	61.56	79.25	92.69	17.16	62.10	72.47	80.28
	90d	19.58	72.06	86.68	95.04	16.56	53.32	61.69	76.62

Note: The decrease rate is the decrease rate in the fixed base ratio method.

admixture of ferronickel slag powder exceeds 20%, the effect of its impermeability is slightly slowed down. At the same time, the erosion from a marine environment has a large negative effect on the impermeability of the soil-cement. However, the impact of the marine environment on the permeability of soil-cement can be alleviated by an increase in the amount of ferronickel slag powder.

- (4) 90d Immersion age: at this time, the impermeability of the soil-cement is greatly improved with the increase of the amount of ferronickel slag powder, and the increase in impermeability of the soil-cement will slow down when the admixture of ferronickel slag powder is high. The impermeability of soil-cement can be greatly improved by increasing the amount of ferronickel slag powder, at the same time, the erosion effects to the soil-cement from being in a marine environment can be reduced. Therefore, the mixing value of mineral admixtures in the chapter meets the standard research range, and the optimum mixing amount of ferronickel slag powder is 40%.



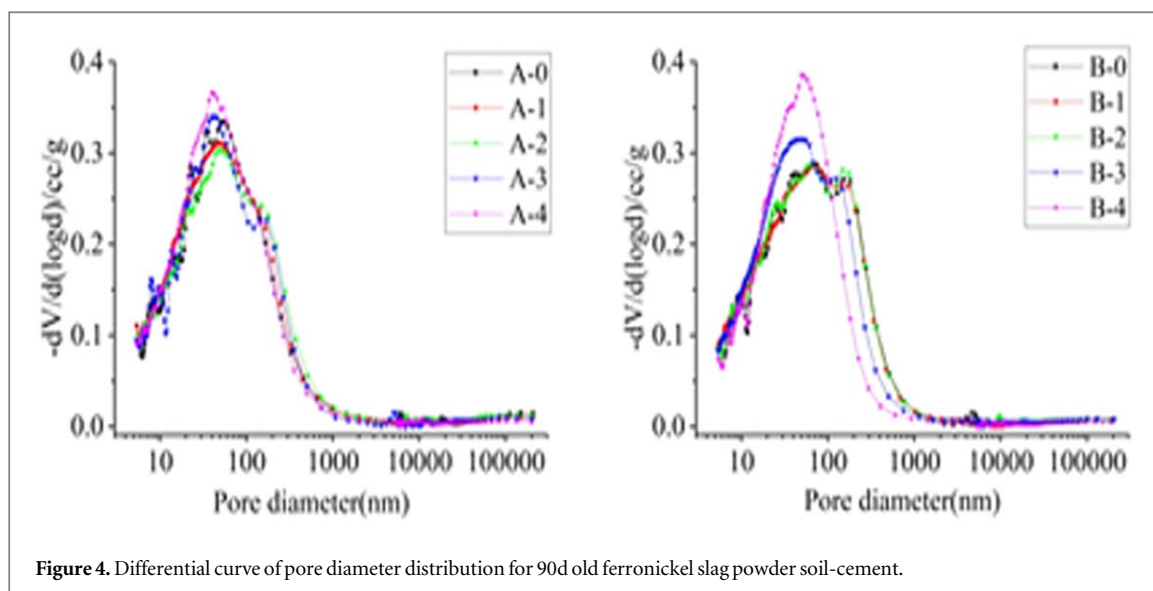


Figure 4. Differential curve of pore diameter distribution for 90d old ferronickel slag powder soil-cement.

### 3. Mercury intrusion test

To better explain the impact of ferronickel slag powder on the corrosion resistance of soil-cement, the Mercury intrusion method was used to test the pore diameter distribution, most probable pore diameter and porosity of the ferronickel slag powder soil-cement and other parameters. The corrosion resistance mechanism of ferronickel slag powder soil-cement was analyzed by evaluating the microscopic pore structure.

The Pore Master 60GT mercury intrusion porosimeter (MIP) was used to perform the tests, to test the pore diameter and distribution of soil-cement. The test included two parts: a low-pressure test and a high-pressure test. The pressure value of the low-pressure pore was 1.03 psi–25 psi, and the measurable diameter of the pore was 750–5  $\mu\text{m}$ . The pressure value of the high-pressure pore was 5 psi–50,000 psi, and the measurable diameter of the pore was 11  $\mu\text{m}$ –3 nm. The soil-cement's rate of hydration is relatively slow, so a 90d old sample with the desired engineering characteristics was selected for the test.

The ferronickel slag powder soil-cement in the clean water environment and the marine environment were subjected to MIP tests for both high and low-pressure pores. The parameters that can characterize the pore structure morphology and the pore diameter distribution of the ferronickel slag powder soil-cement were collected by a computer. The differential curve of the pore diameter distribution of soil-cement with different admixture content of ferronickel slag powder is shown in figure 4.

'Total porosity' can be expressed by the differential curve obtained by MIP. The area enclosed by the differential curve graph and the horizontal axis is the total porosity of the soil-cement. The larger the peak value of the differential curve in a certain range is then the larger the pore volume of soil-cement in this range will be. The 'most probable pore diameter' is the pore diameter with the greatest occurrence probability in the soil-cement. The pore diameter corresponding to the peak in the differential curve is the most probable pore diameter, and the characteristics of the porosity and the pore diameter distribution of soil-cement can be represented by its size. Therefore, the most probable pore diameter is one of the important factors affecting the impermeability of soil-cement.

It can be seen from figure 4 that at 90d old, the hardening effect of the cement inside the soil-cement stabilizes, and the development trend of the differential curve of the soil-cement pore diameter under the clean water environment is virtually the same. The most probable pore diameters of soil-cement with a content of 0% to 40% ferronickel slag powder are 60.73 nm, 51.41 nm, 47.44 nm, 42.71 nm, and 39.79 nm, indicating that its impermeability increases with the admixture of ferronickel slag powder. Although the hydration at 90d old tends to be complete, the active effect of the ferronickel slag powder inside the soil-cement still plays a larger role. Therefore, when the admixture of ferronickel slag powder is 40%, the most probable pore diameter is reduced by 34.5% compared with the baseline group. In the marine environment, the most probable pore diameters of the soil-cement of each mixing ratio were 68.42 nm, 66.72 nm, 61.52 nm, 51.76 nm, and 49.73 nm, respectively. Compared with the clean water environment, the soil-cement with the same ratio had a larger increase in the most probable pore diameter. At this time, the most probable pore diameter of the soil-cement with 40% ferronickel slag powder was still the smallest, which is consistent with the results of the better impermeability of soil-cement when the 40% ferronickel slag powder is mixed in the previous section. At 90d old, the ferronickel slag powder mixed into the soil-cement plays an increasing role in its strengthening. The glass body in the

ferronickel slag powder has a similar pozzolanic effect in the soil-cement, which can generate hydration products to make the structure of the soil-cement more compact. When soil-cement is directly exposed to the marine environment, the corrosive substances in the environment (mainly  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$ ) have a greater corrosive effect on soil-cement and degrade its performance.

At 90d old, the total porosity of soil-cements A-0, A-1, A-2, A-3 and A-4 in a clean water environment were 36.47%, 34.28%, 33.68%, 32.22% and 31.19%, respectively. The total porosity of soil-cements B-0, B-1, B-2, B-3, and B-4 in the marine environment were 52.89%, 52.43%, 41.23%, 36.52%, and 34.81%, respectively. The total porosity of soil-cement decreases with the admixture of ferronickel slag powder, and the erosion of the marine environment causes the increase of cement porosity. This is because the ferronickel slag powder mainly plays a micro-aggregate effect and an active effect in the soil-cement. The porosity of soil-cement can be effectively reduced by mixing ferronickel slag powder and the soil-cement can be made to form a more compact structure. The integrity of the soil-cement can be improved while also alleviating the erosion caused by the marine environment.

## 4. Conclusions

In this paper, soil-cement mixed with ferronickel slag powder was subjected to permeability tests, and mercury intrusion tests were performed on the soil-cement immersion curing in the clean water and marine environments. The impact of different curing environments, different admixture contents of ferronickel slag powder, and different immersion ages on the impermeability of soil-cement were studied, and the following conclusions were drawn:

- (1) At the 7d and 28d old, the difference in permeability coefficient between the clean water environment and the marine environment of the soil-cement is not large, and even the value of the permeability coefficient at 7d is basically the same, showing that the marine environment has little effect on the permeability of the ferronickel slag powder soil-cement before 28d old.
- (2) The permeability coefficient of soil-cement can be reduced by using an admixture of ferronickel slag powder in clean water and marine environments, thus improving its impermeability. With the increase of the admixture of ferronickel slag powder, the impermeability coefficient of the soil-cement gradually decreases. However, when the admixture of ferronickel slag powder exceeds 20%, the enhancement effect of soil-cement impermeability slows down; namely, the constant increase of ferronickel slag powder has a smaller impact on the impermeability of soil-cement.
- (3) The permeability coefficient of the marine environment is clearly greater than that of the clean water environment. The deterioration of the impermeability of soil-cement by the marine environment can be slowed down by increasing the admixture of ferronickel slag powder in soil-cement. In the tests, the range value of the admixture content of ferronickel slag powder was selected based on the specification, and the optimum content of ferronickel slag powder obtained in the study was 40%.
- (4) It can be seen from the results of the Mercury intrusion test that the ferronickel slag powder mixed into soil-cement could exert a micro-aggregate effect and an active effect. With the increase of the admixture of ferronickel slag powder, the most probable pore diameter of the soil-cement gradually decreased, and the total porosity gradually decreased. The soil-cement matrix was more compact and an optimum mixture was achieved when the admixture was 40%. Therefore, the deterioration of pore structure in the marine environment can be significantly alleviated and an improved impermeability can be attained by the admixture of ferronickel slag powder.

## Data availability statement

No new data were created or analysed in this study.

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