Article

Experimental Study on the Strength and Durability for Slag Cement Mortar with Bentonite

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Abstract: This study focused on improving the durability of concrete infrastructures located in marine environments. When structures made of concrete are exposed to a marine environment, the combined effects of mechanical and chemical actions, such as attacks from sulfate and chloride, may deteriorate the material, then cause corrosion of the embedded reinforced steel bars, and finally result in shortening of the service life of the structure. Therefore, proposed in this work is a method mainly focused on improving the strength and durability of concrete composed with slag cement as a binder, which due to its pozzolanic reaction is commonly applied in construction projects in marine environments. The durability of slag cement mortar can be improved through the application of bentonite material by utilizing bentonite's very finely divided form and its reactivity to sulfate so that the larger capillary pores in concrete can be filled. It was found that a slight (approximately 0.25%) replacement of slag cement with bentonite material effectively improved the durability of the slag cement mortar in terms of moisture absorption, compressive strength, and weight loss after a sulfate resistance test and the evaluation for chloride permeability.

1. Introduction

It is quite common that concrete structures are located in environments full of pollutant ingredients and chemicals that are harmful to concrete, such as sewer systems, water processing plants, and infrastructure located in marine environments. Maintaining good durability for the structures located in such environments always imposes serious challenges. After physical erosion and mechanical loading, chemical attacks usually are the next main cause of damage to concrete material. After the deterioration of material, such as by its neutralization, the corrosion in ferro-material will further advance due to the presence of sulfate, chloride, and acidic elements in the environment that may combine and worsen the deterioration of the material [1–3].

The loss of impermeability of concrete material is usually a major problem that causes damage to the concrete structures located in aqueous environments. The neutralization of concrete is usually caused by the permeation of chloride, which gradually deteriorates the concrete, damages its impermeability, and then reduces its strength. Once the water seeps into the material along with oxygen, corrosion occurs on the steel reinforcements. This phenomenon has been reported for decades [4–6] especially for structures located in aqua-environments.

For the reinforcement concrete structures located in aqua-environments, the damage of the structure and material can also be a result of combined effects of the chemical actions of seawater constituents on cement hydration products, crystallization pressure of salts within concrete, corrosion of embedded steel in reinforced or pre-stressed steel cable members when they are exposed to hazardous ingredients, and physical erosion due to the action of waves and floating objects. The corrosion in reinforced steel bars embedded in concrete is

mostly due to the intrusion of sea water and oxygen, which penetrates through the small cracks of the deteriorated concrete [7]. Some other factors such as sulfate, chloride, and acidic elements can also worsen the problem. It is important to protect the structures made of reinforced concrete in aqua-environments from damage resulting from the deterioration of concrete and corrosion of steel. Among various methods, the most effective is the improvement of the impermeability of the concrete material. At the same time, the other positive properties of concrete materials, such as their strength, must not be influenced.

One of the methods to improve the impermeability of concrete material is the application of pozzolanic material [8,9] because of its finely divided form (less than 75 μ) and its chemical reactivity with calcium hydroxide when moisture is present. In this way, the larger capillary pores in the mixture are filled and, accordingly, we improve the impermeability of concrete material. Therefore, concrete constructions located in marine environments using slag-cement as binders are popular due to the pozzolanic reactions of the slag material [10–12]. However, in order to examine if further improvement to the durability of slag cement concrete located in marine environments is possible, a bentonite material was applied in this study.

Bentonite has been a renowned material, widely applied for masonry, grout, and sludge solidification works due to its good properties of having fine grains, great moisture absorption capability, and bulking properties when wet. The application of bentonite clay for the stabilization and solidification of sewage sludge containing heavy metals has a very good effect on impermeability [13]. A similar application for permeability reduction to the cut-off wall of dams was also reported [14], where part of the cement was replaced by a bentonite material in the plastic concrete that has a low elastic modulus compatible with the surrounding soil. Other applications for bentonite are also under development, such as a study of the light-weight aggregates made from fly ash, where the bentonite was used as a binder material when heat treatment process was applied [15].

In a study of concrete with replacement of cement by bentonite [16], it was found that the water demand in concrete was increased while the compressive strength was reduced with respect to the replacement ratio of bentonite. However in Ahmad's study [16], the replacement ratio of cement to bentonite was high, from 20% to 50%, and therefore, it is only suitable for concrete with sufficient strength for low-cost construction. A similar finding for concrete with replacement of cement with bentonite but at a lower ratio of replacement (at most 21%) was presented [17], where they also found that the workability and water absorption decreased with increased bentonite content. In a later study, the waterproofing performance of cement mortar containing bentonite can reached a maximum impermeability pressure 2.37 times that of the reference group [18]. Other applications of concrete combined with bentonite and with other materials like polymer balls or recycled aggregates have also been found [19,20]. It was found that the mechanical and microstructural properties of such concretes combined with bentonite were comparable to that of plain concrete.

In terms of compressive strength, bentonite was also used for high-strength concrete [21], and it was concluded that bentonite can be used in concretes where later-stage strength is required because bentonite results in poor early-stage compressive strength but gives good results in later stages. The authors of [17] and [22] also reported that some strength gain was found with longer curing times, which is a very encouraging finding. Therefore, we aimed to take advantage of bentonite to improve the impermeability properties and find the variation of material characteristics in slag cement. By replacing a small amount of binder (cement and slag) with bentonite material, the investigation focused on the improvement of impermeability and durability for slag cement mortar. The purpose of this study was the improvement of the durability of concrete by utilizing slag cement as a binder in costal and marine environments rather than a cost-saving application by consuming large amounts of bentonite material.

In this study, various levels of binding material in a slag cement mortar were substituted by bentonite. This is an alternative scheme to improve the impermeability of the

concrete, and because of that improvement, it is also expected to provide better capability for sulfate resistance and adsorption ability for chloride, so that eventually the durability of the concrete material can be improved. During the proportioning design, several water/binder ratios were proposed, and laboratory tests were performed to examine the influence of the addition of the bentonite material on both the basic properties and the enhanced characteristics of the slag cement mortar. To determine the durability of the bentonite-added slag cement mortar, tests of its sulfate resistance capabilities were further performed, where the weight loss, moisture absorption, and penetration of chloride were measured. A series of compressive strength tests were also carried out for the specimens subjected to sulfate corrosion and those not subjected to sulfate corrosion.

2. Materials and Proportioning Design

2.1. Cement Slag and Fine Aggregate

The cement utilized in this study was Type-I produced by the Taiwan Cement Company, and the related properties of the applied cement complied with the ASTM C150 [23] specifications and are shown in Table 1. The slag was produced in the blast furnace at the China Steel Corporation in Taiwan. Both the physical properties and the chemical compounds of the slag are also shown in Table 1, and they conformed to CNS12549 specifications [24]. The fine aggregate utilized in this study conformed to the specifications of ASTM C33 [25] for concrete material.

Table 1. Properties of the slag and cement used in this study.

Sample	Physical Properties					
	Specific Surface	Sieve #325 (Retained %)	Specific Gravity	Reactive Index (%)		Air Content (%)
				7-day	28-day	
Cement [1]	-	5.90	3.14	-	-	-
Slag	440	1.10	2.89	88.1	119.2	3.23

Sample	Chemical Properties								
	Compound (wt%)								Basicity [4]
	LOI [2]	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	SO_3	S [3]	
Cement [1]	3.00	20.70	5.40	3.20	2.00	63.90	2.00	3.50	-
Slag	1.43	33.47	14.79	0.41	6.11	41.61	6.11	0.59	1.87

[1] Cement was Type-I Portland cement. [2] Loss on ignition. [3] Sulfide and sulfur. [4] Basicity = $\frac{Al_2O_3+CaO+MgO}{SiO_2}$.

2.2. Bentonite Material

The material of bentonite used in the study was purchased from the local industry. The properties of the bentonite complied with API-13A (American Petroleum Institute) [26]. The basic properties of the bentonite used in this study are listed in Table 2. It can be noted from Table 2 that the particle size of bentonite is very fine, such that 96% by weight of the bentonite material passes a screen of 75 μm. The specific density of the bentonite is almost the same as that of cement. Therefore, the advantage of physical property similarity is useful for the partial substitution of the cement material by bentonite.

2.3. Proportional Design of Specimens

The base materials of the mixture of cement mortar were similar to that of a traditional cement mortar, which includes Type-I cement, sand as the fine aggregate, and blast-furnace slag. All materials other than the sand, namely, cement, slag, and bentonite, are binding materials that are hydrated with water. The water-binder (w/b) ratios were designed as 0.445, 0.485, and 0.550 and the corresponding proportion of the fine aggregate, sand remained the same. Due to the fact that bentonite has good moisture absorption capacity, the proposed water-binder ratio was based on a range above the water-binder ratio of a standard proportion.

Table 2. Material properties of bentonite.

Physical Appearance	Light Tan/Gray-Green Powder
Specific gravity	2.3~2.6
Bulk density	48~52 (lb/ft^3), 769~833 (kg/cm^3)
Viscometer dial reading at 600 rpm	Min: 30
Yield point/plastic viscosity ratio	Max: 3
Filtrate volume	Max: 15 cm^3
Residue >75 micron (wet screen)	Max: 4% wt
Moisture	Max: 10% wt
Suspension of 22.5 grams into 350 cm^3 water	

According to the test proposal, part of the binding material was substituted for the bentonite and, therefore, the same proportions of both the cement and slag were substituted by the same amount of bentonite. The ratios of the substitutions were 0.25%, 0.50%, and 0.75% in terms of the ratio of the weight of slag and cement while a reference group without substitution was also tested. A total of 12 sets of mortar specimens were tested and the detailed proportion in percentage of weight of individual materials are presented in Table 3.

Table 3. Proportional design for the materials applied.

Specimen Code	Cement (kg)	Slag (kg)	Bentonite (kg)	Water (kg)	Sand (kg)
0BT445	0.2500	0.2500	0		
25BT445	0.2498	0.2498	0.00125		
50BT445	0.2493	0.2493	0.00250	0.223	1.375
75BT445	0.2483	0.2483	0.00375		
0BT485	0.2500	0.2500	0		
25BT485	0.2498	0.2498	0.00125		
50BT485	0.2493	0.2493	0.00250	0.243	1.375
75BT485	0.2483	0.2483	0.00375		
0BT550	0.2500	0.2500	0		
25BT550	0.2498	0.2498	0.00125		
50BT550	0.2493	0.2493	0.00250	0.275	1.375
75BT550	0.2483	0.2483	0.00375		

Note for coding: XXBTXXX; The first set of numbers is for the percentage of bentonite: 50BT—0.50% bentonite. The second set of numbers is for the ratio of the water/binder (w/b): 50BT445—0.445 w/b.

3. Proposed Experimental Tests

The basic properties, especially the consistency of the fresh mixture for the slag cement with bentonite, are important because of the better absorption capacity of bentonite. Therefore a flow-table test (ASTM C230) for the fresh mortar and the setting time (ASTM 191 and 266) were both examined for fresh mixtures of slag cement mortar [27]. Figure 1 shows the testing result for the flow-value corresponding to the replacement ratio of bentonite. It was found that the flow-value drops with respect to the increment of the substitution ratio of bentonite. However, the flow-values for all of the test specimens were still high. The results of tests showed that both the readings of the flow table test and setting time [27] complied with the ASTM standard. For the properties of hardened specimens, the capacity of moisture absorption based on ASTM C642-90 was performed to test specimens that were cured in the lime water for 28 days. A series of standard compressive strength tests (ASTM C109) were also carried out for the slag cement mortar with various substitution ratios of bentonite for the binding material. The water/binder ratios were also taken into account during the compressive strength tests. The tests of

compressive strength were performed on the specimens cured for both 7 days and 28 days to examine the early strength.
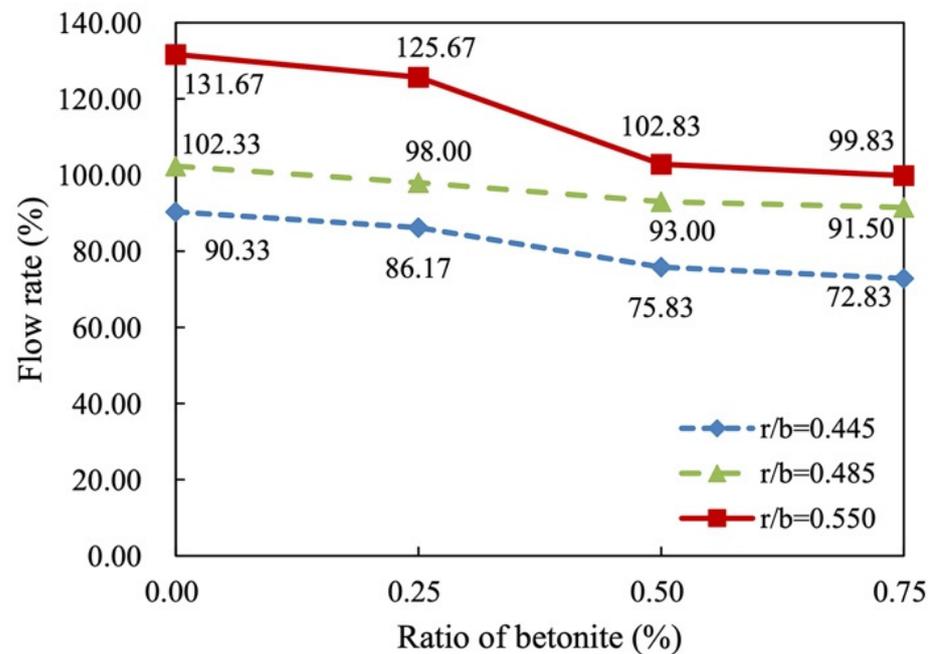

Figure 1. Flow-values corresponding to the replacement ratio of bentonite [27].

The micro-morphological characters of each concrete specimen were determined using scanning electron microscopy (SEM, S-3000N, Hitachi, Tokyo, Japan), a powerful analytical technique for material characterizations. Before the observation, each sample was dried in a conventional oven over 24 h until the weight was stable to avoid residual water existing in the voids. To provide an electronic conductive surface for higher quality SEM images, fine gold particles were sputtered on the specimen surface using a sputter coater (E-1010, Hitachi, Tokyo, Japan). All preparations were based on the guidelines of ASTM C1723-16 [28]. SEM images captured using an accelerated voltage of 15 kV and were characterized by the secondary electron mode, which benefited the inspection of the topography of the surface.

To determine the effect on durability, such as the resistance to chloride compounds due to the decrease in permeability, the absorption of the chloride by the bentonite, and the variation of the properties of the hardened specimens after suffering sulfate attacks, sulfate resistance tests and the chloride-blocking tests (ASTM C1202-19 [29]) were also carried out. Similarly, specimens of three various water/binder ratios and four replacement ratios of bentonites were soaked in the solution of sodium sulfate and then dried out, alternately, for five cycles (ASTM C1202). After the sulfate resistance testing, examinations of the shape distortion and volume variation of the specimens were performed. In addition, the possible weight loss of each specimen was measured, and the increment of moisture absorption rate was also recorded. All of these data were compared to the data before the sulfate resistance test, which combined indicate the durability of the slag cement mortar against sulfate attack. A series of compressive strength tests were also carried out for the specimens subjected to sulfate corrosion. The test results were compared to the strength obtained from the specimens that were not subject to sulfate corrosion and were cured for 28 days in a standard procedure.

4. Results and Discussion

4.1. Compressive Strength Development before the Sulfate Resistance Test

The development of the compressive strength for the slag cement mortar cured in lime water for 7 days or 28 days with various substitution ratios of bentonite in the binding

material are presented in Figure 2, where (a), (b), and (c) correspond to the 0.445, 0.485, and 0.550 water/binder ratios, respectively. The blue colored bars are for the specimens cured for 7 days while the grey bars are for the specimens cured for 28 days. The progress of strength development can be clearly observed from these figures. It can be noted from these test results that 70~74% of the compressive strength was developed for the specimens without bentonite cured for 7 days. For the specimens with partial replacement of bentonite for the slag cement in the group of 0.458 and 0.550 w/b ratios as shown Figure 2b,c, a range of 70~76% compressive strength was obtained for the specimens cured for 7 days. An interesting phenomenon was observed is that for the specimens with bentonite and higher w/b ratios (e.g., w/b = 0.550) the compressive strength developed earlier, where the strength developed in 7 days reached as high as 78% for the specimen with 0.75% replacement of bentonite.
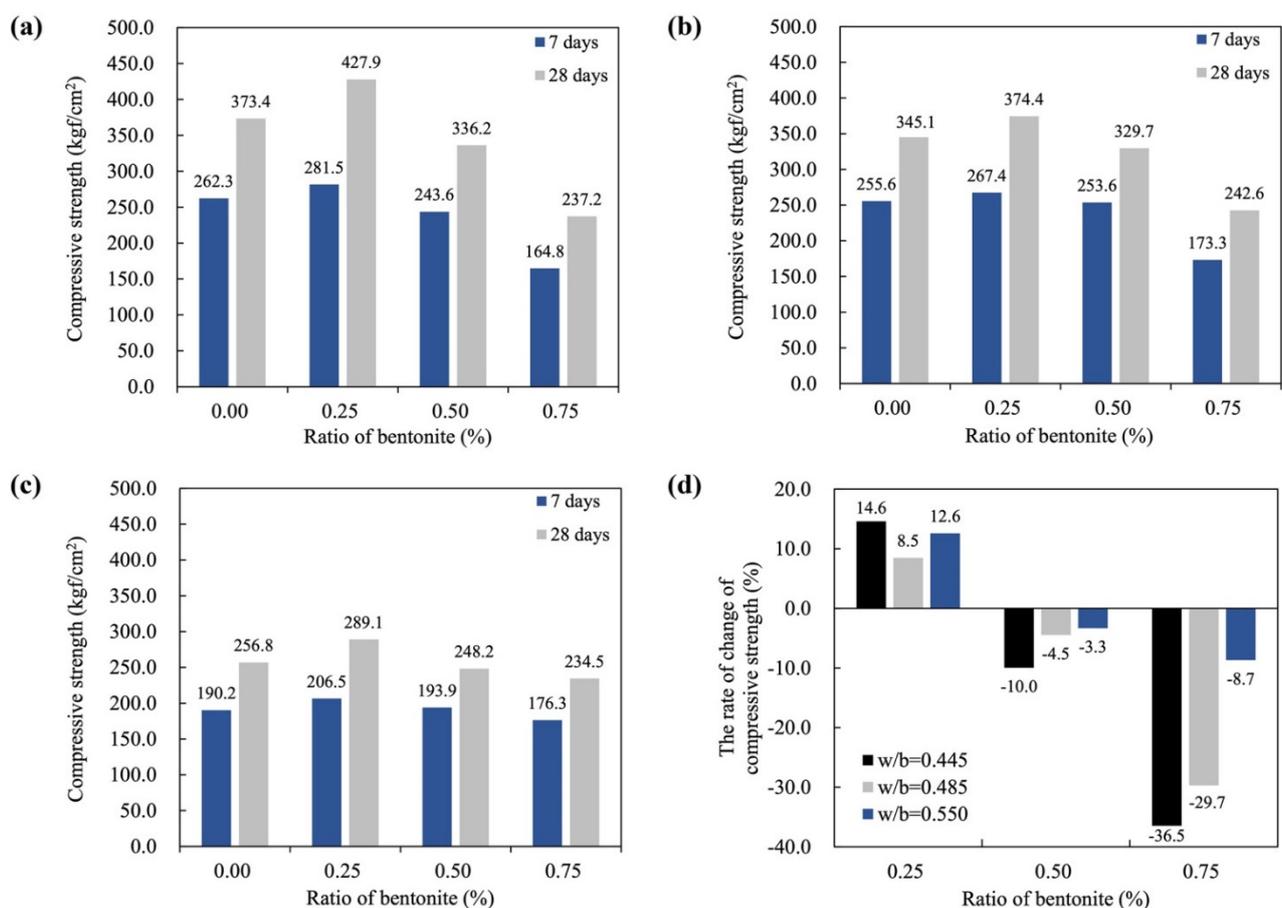

Figure 2. Compressive strength for various replacement ratios of bentonite. The gray colored column is for the specimens cured for 7 days while the navy column is for the specimens cured for 28 days: (**a**) w/b = 0.445, (**b**) w/b = 0.485, and (**c**) w/b = 0.550. (**d**) Variation rate of compressive strength corresponding to the replacement ratio of bentonite.

One special phenomenon observed was for the case of 0.25% replacement of bentonite, which had the highest compressive strength at both 7 days and 28 days of curing but also had the slowest strength development at 7 days of curing, as presented in Figure 2a, where only 65.8% compressive strength was developed for the specimen of 0.25% replacement of bentonite after 7 days curing. The strength development at 7 days of curing for the specimen of 0.25% replacement was also the lowest (71%), as shown in Figure 2b,c compared to that of specimens without bentonite or with higher replacement rations of bentonite (i.e., 0.50% and 0.75%).

Figure 2d shows the variation rate of the compressive strength for the slag cement mortar cured for 28 days when various ratios of binders (cement and slag) were replaced

by bentonite. For the case of 0.25% replacement of bentonite in various water/binder ratios, the compressive strength of the mortar increased, where for the 0.445 w/b ratio, the increment in the compressive strength of the mortar was about 15% higher compared to that of the specimen without bentonite. For the strength performance of specimens with higher replacement ratios of bentonite (other than 0.25), the compressive strength seemed to decrease, corresponding to the increase of the replacement ratios of the bentonite. The decreasing trend of the strength corresponding to the increase of replacement ratios of bentonite seems to be mitigated when the w/b ratio is high, such as the case of w/b = 0.55.

4.2. Weight Loss after the Sulfate Resistance Test

The losses of weight for the specimens with various replacement ratios of bentonite subjected to sulfate resistance testing are shown in Figure 3a–c for specimens with various water/binder ratios. As shown in Figure 3, corresponding to the increased number of immersive cycles of specimen in the solution of sodium sulfate, the weight loss of most specimens increased. In the first cycle, the weight slightly increased due to the attachment of the sodium sulfate reaction products on the specimen. As can be seen in Figure 3a, where the w/b ratio is 0.445, partial replacement of slag cement with bentonite seems to reduce the loss of weight of a specimen after being subjected to the sulfate resistance test. The higher is the replacement ratio, the smaller is the weight loss. However, for the group with 0.25% replacement of bentonite, the weight loss phenomenon was similar to that of the sample without replacement with bentonite. When we examine Figure 3b,c, we can see that when the w/b ratios are higher, the reduction of weight loss due to partial replacement of slag cement by bentonite is not significant, especially when the w/b ratio is 0.55.
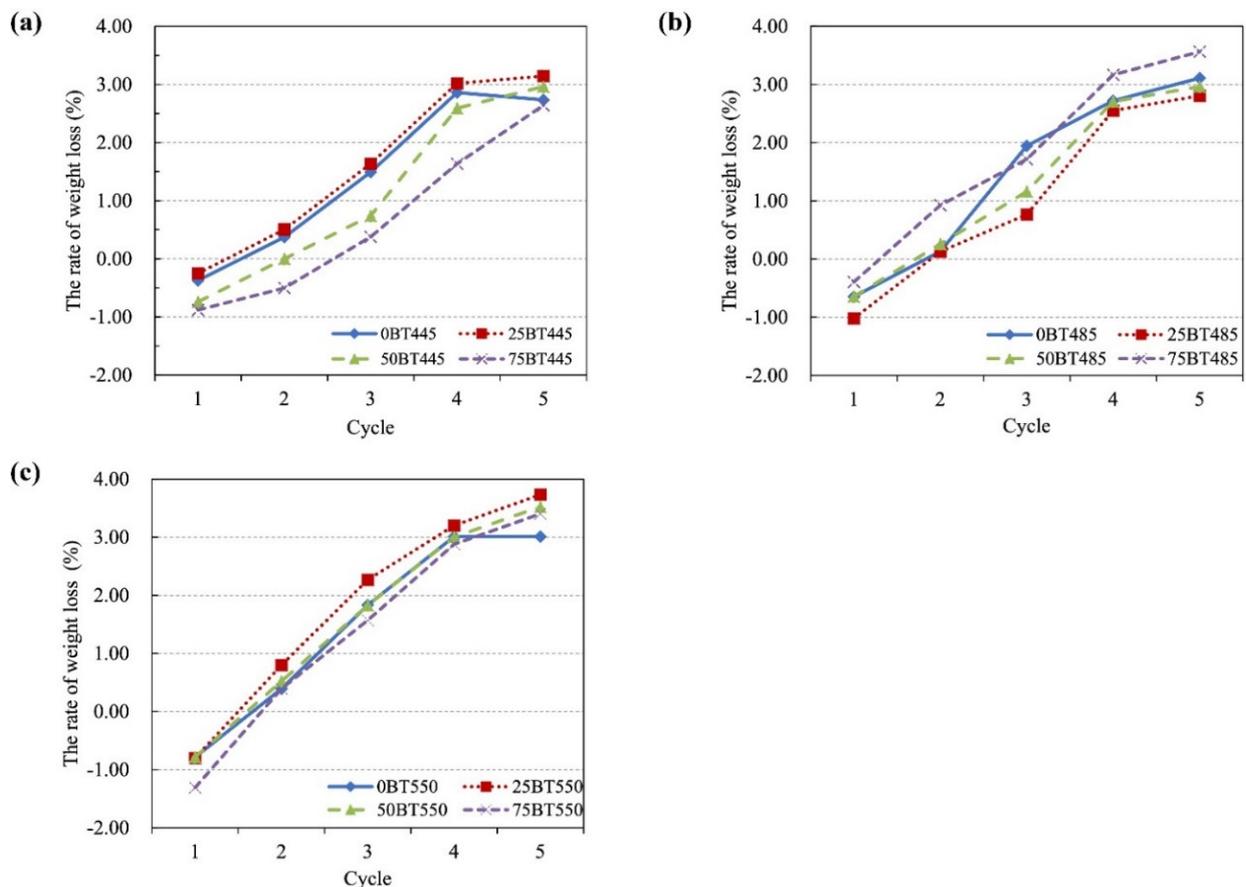

Figure 3. Ratio of weight loss after the sulfate resistance test: (**a**) w/b = 0.445, (**b**) w/b = 0.485, and (**c**) w/b = 0.550.

4.3. Variation of Moisture Absorption after the Sulfate Resistance Test

The moisture absorption capacity of specimens after being subjected to the sulfate resistance test was evaluated and the results are shown in Figure 4, where most values of moisture absorption were well over 10%. The variation rate of moisture absorption capacity for the specimens subjected to the sulfate resistance test is also interesting when compared to the data for the specimens that were not subjected to sulfate sodium solutions.
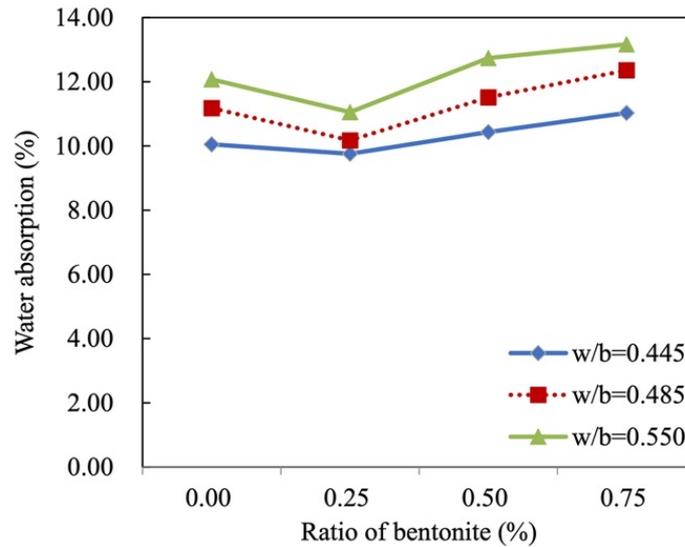

Figure 4. Absorption capacity after the sulfate resistance test for various ratios of bentonite.

Figure 5 shows the variation in the rate of moisture absorption capacity in terms of percentage relative to specimens that were not subjected to the sulfate resistance testing. Generally, the absorption capacity was increased for specimens subjected to the sulfate resistance test due to the erosion effect of the chemical reactions on the surface of the specimens. However, for the group of 0.25% replacement ratio of bentonite to slag cement, the absorption capacity was reduced for every w/b ratio compared to specimens without bentonite replacements. The variation rate of absorption capacity was reduced as low as 3.58%, when water/binder ratio was 0.55.
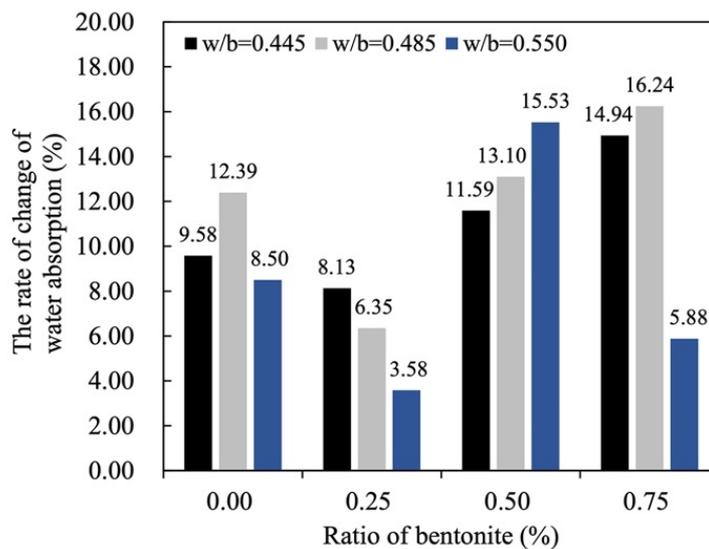

Figure 5. Variation rate of absorption capacity after the sulfate resistance test.

4.4. Variation of Compressive Strength after the Sulfate Resistance Test

The compressive strengths for the specimens subjected to the sulfate resistance capability test were also investigated and the results are shown in Figure 6a–c for specimens with various w/b ratios. Each ratio of bentonite replacement has two columns to represent the compressive strength acquired before and after the sulfate resistance test. Figure 6 shows the trend of the compressive strength corresponding to the replacement ratio of bentonite and the reduction phenomenon of strength due to reactions of the sodium sulfate solution. The phenomenon of strength reduction was found for all specimens subjected to sulfate resistance testing in three different w/b ratios. The reduction rate of strength was as high as 30%, such as that of the 0.485 w/b ratio without bentonite replacement. The phenomenon of strength reduction was mitigated corresponding to the increase of the replacement ratio of bentonite, as observed from specimens with 0.445 and 0.485 w/b ratios. In the extreme case with the 0.75% replacement ratio of bentonite, the reduction rate of the compressive strength of specimens subjected to sulfate corrosion was decreased by as much 1% compared to that of specimens with a 0.445 w/b ratio. Therefore, the usage of bentonite can effectively reduce the strength-reduction phenomenon if proper replacement ratios for the bentonite are applied. Generally, the mitigation effect of strength reduction of slag cement mortar due to sulfate attack was more significant for the specimens with lower w/b ratios (i.e., 0.445 and 0.485) and higher replacement ratios of bentonite (i.e., ratio of 0.75).
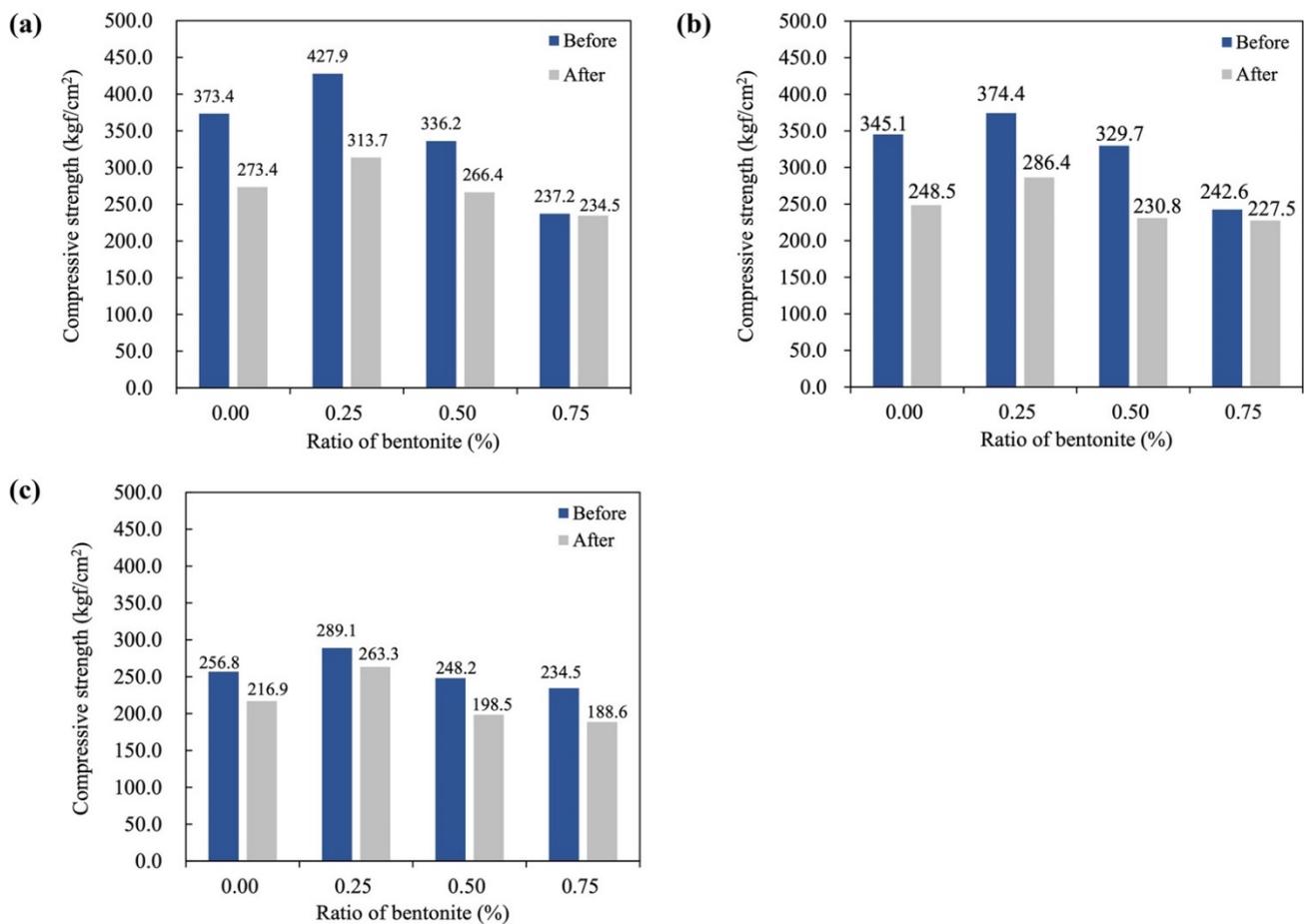

Figure 6. Comparison of compressive strength before/after the sulfate resistance capability test: (**a**) w/b = 0.445, (**b**) w/b = 0.485, and (**c**) w/b = 0.550.

4.5. Results from Rapid Chloride Permeability Testing

A standard test based on ASTM C1202-97 using electrical indication to examine the chloride ion penetration ability was performed. Figure 7 shows the testing results, where the chloride penetration ability in terms of coulombs for specimens without bentonite was generally higher than 20 mA, and the detected current stayed at a constant level. However, when the bentonite was replaced at 0.25%, data from the electrical current indicated that the chloride penetration ability was significantly reduced, where the chloride was reduced by about 97%. The mitigation of the penetration ability of chloride from partial substitution of binders in the slag cement by bentonite is very significant and obvious.
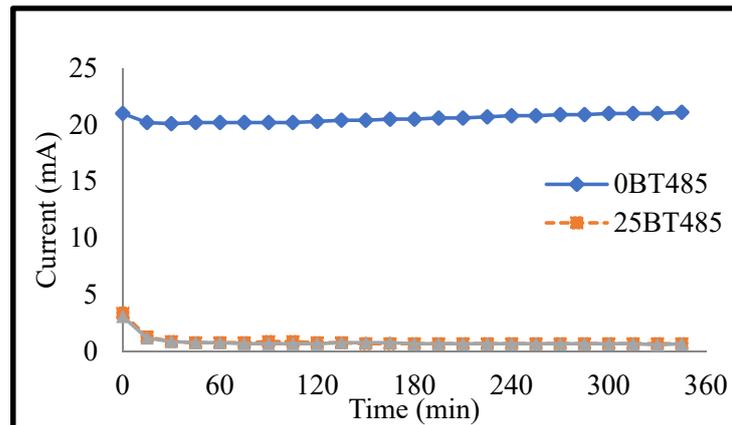

Figure 7. Chloride permeability testing results for bentonite mortar.

4.6. Microstructures of Slag Cement Mortar with Bentonite

The typical structures of bentonite are presented in Figure 8, where the enlargement of the structure in the picture is 2000 times. Figure 9a,b depict typical structures of a hydrated slag cement mortar with bentonites, where the lumped bentonite(B) constituents can clearly be observed along with the hydrated products of slag cement such as the needle-shaped hydrated sulfate-aluminate silicate (ettringite, AFt) and C-S-H gel [30]. Figure 9a shows a uniform distribution of bentonite deposited in every position, meanwhile, more voids exist with the higher addition of bentonite in the mix, as presented in Figure 9b. Figure 9c,d show the microstructures of hydrated slag cement mortar with bentonite after performing the sulfate resistance test. In both pictures, some crystallized sodium sulfate(S) can be found among the C-S-H colloid. Moreover, some gypsum (G) plates, a compound of sulfate and calcium cation, are observed on the sample [31].
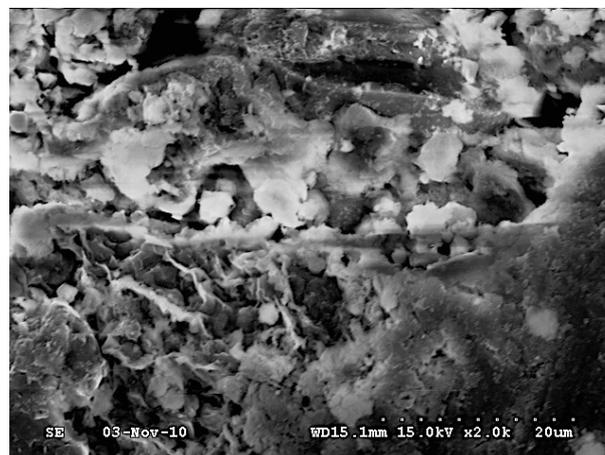

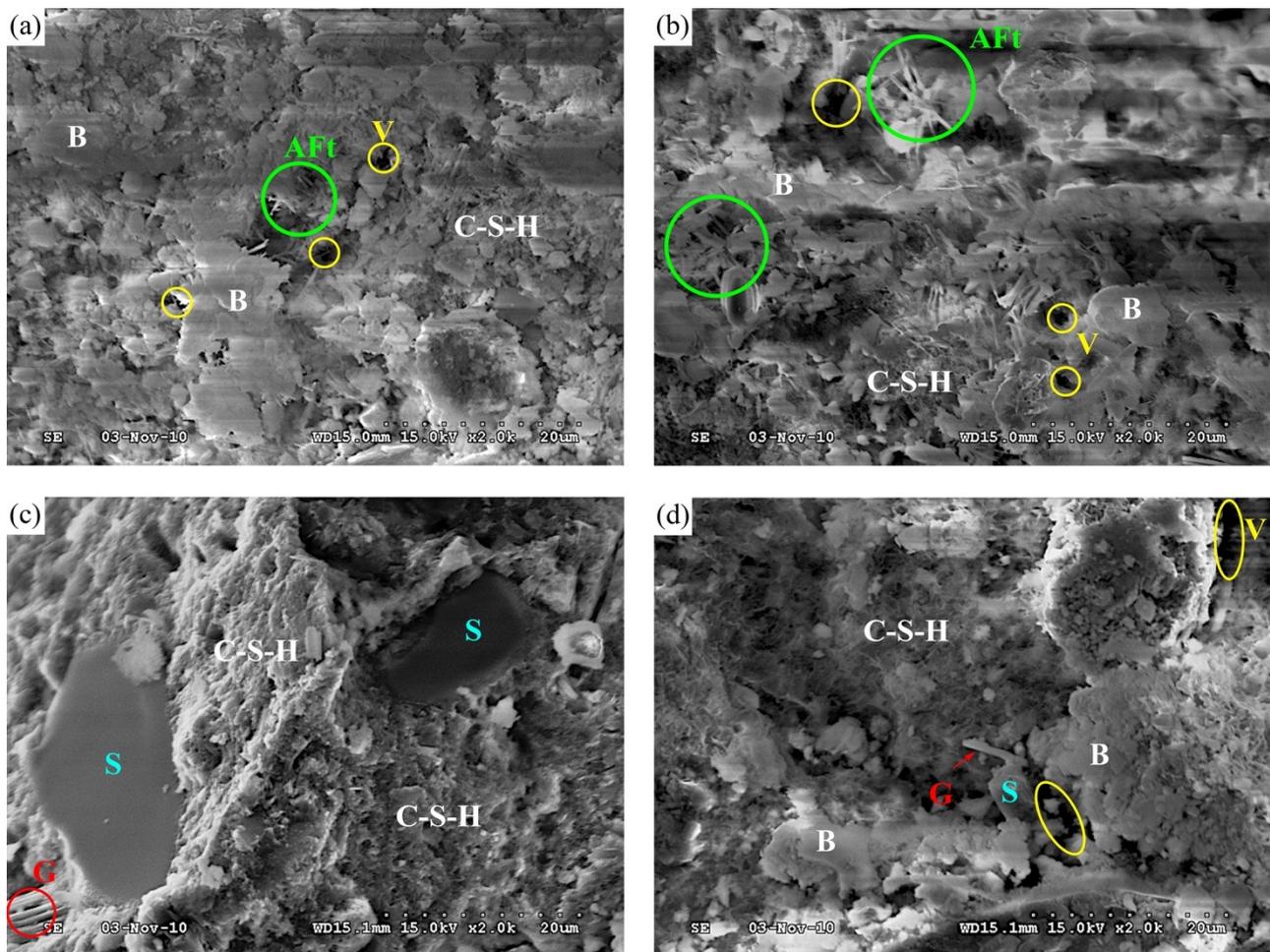**Figure 8.** Typical structure of bentonite material.

Figure 9. SEM images of cement mortar with 0.25% bentonite (**a**) before and (**c**) after the sulfate test; SEM images of cement mortar with 0.50% bentonite (**b**) before and (**d**) after the sulfate test.

The lower water absorption and the higher compressive strength were obtained at an additional level of 0.25% bentonite in each w/b ratio. However, impermeability and strength of mortar decreased as bentonite replacement ratio increased over 0.25%. Due to the fact that particles of bentonite are much finer compared to most aggregates, they are uniformly arranged in the binder matrix of concrete and make the material denser. The bentonite particles, with high specific surface area, result in an high amount of water moistening of their substantial surface areas. This phenomenon limits the pozzolanic material hydration reaction, and then that diminishes the workability and impermeability of high bentonite content mixes. The pozzolanic reaction could also have caused the formation of extra calcium silicate hydrate products that decreased the voids volume. Therefore, the mixes incorporating suitable content of bentonite are able to improve the mechanical properties and impermeability, which is in line with the finding of SEM as well as that of a previous study [32].

5. Conclusions

An experimental study for slag cement mortar with partial substitution of binders by bentonite material was carried out in this study. Both properties of fresh mixtures and the hardened specimens were examined experimentally. Based on the testing results and the corresponding discussions, some valuable conclusions are drawn as follows.

1. The capacity of moisture absorption for the slag cement mortar may be influenced by the increase of the substitution ratio of the bentonite to the binders in the mortar,

but not significantly. However, the increase of w/b ratio may cause a significant increment of moisture absorption of the mortar for most cases.

2. The moisture absorption capacity is increased for the slag cement specimen subjected to the sulfate resistance test due to the erosion effect of the chemical reactions on the surface of the specimens. A replacement of a small amount of the binder in the slag cement mortar by bentonite may improve the moisture absorption, such as was the case of 0.25% replacement, where the moisture absorption was reduced for every w/b ratio compared to specimens without bentonite replacements.

3. The addition of bentonite material in slag cement mortar may influence the compressive strength. For an appropriate addition of bentonite to the slag concrete, the strength can be upgraded by as much as by 14.6%. However, if the replacement ratio of the bentonite is higher than 0.25%, the compressive strength of the mortar will be reduced corresponding to the replacement ratio of bentonite.

4. For the variation of compressive strength after the sulfate resistance test, the usage of bentonite can effectively reduce the strength-reduction phenomenon for the slag cement mortar. Generally, the mitigation effect of strength reduction of slag cement mortar due to sulfate attack was more significant for the specimens with lower w/b ratios (0.445 and 0.485) and higher replacement ratios of bentonite (0.75).

5. After the sulfate resistance test, the weight-loss of the slag cement mortar specimens due to the sulfate reactions can be improved by the replacement of a small amount of the binder by bentonite when the w/b ratio is smaller (0.445 in this case). However, if the w/b ratio is higher, the mitigation of weight loss is not very significant.

6. The chloride penetration to the slag cement mortar can also be effectively reduced with the application of bentonite.

Therefore, it is concluded that a slight replacement, such as 0.25%, of slag cement with bentonite material may effectively improve the durability of the slag cement mortar in terms of the moisture absorption, compressive strength, and weight loss after the sulfate resistance test and the chloride permeability test.

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