Investigation on the Properties of Carbonated Basic Oxygen Furnace Slag

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ABSTRACT

BOF slag with CaO content of up to 38-50 percent by weight (wt%) caused by a high pH value and expansion ratio, thus restricting the application of the BOF slag, especially for making powdered BOF slag (diameter, $d_p < 4$ mm). Carbon dioxide reduction is another important issue for the steelmaking industries. This study applied BOF slag to capture and reduce the CO₂ emission in the flue gas to both broaden the application of powdered BOF slag through CO₂ reduction. A lab-scale fixed bed carbonation system was installed for this study. The experimental parameters including amounts of water feed, CO₂ concentration, gas flow rate, and carbonation time were investigated. Experimental results indicated that the pH value of the raw and carbonated BOF slag were all less than 12.5. The pH value of the carbonated BOF slag in this study was near 12. The immersion expansion ratio of the powdered BOF slag decreased from 10.1 to 2.1%. In addition, the maximum achievable compression strength of the carbonated BOF slag in this study was approximately 36.72 kg/cm². Moreover, the results from the dissolution test of BOF slag, just slightly higher than that without BOF slag. The concentrations of Cr, Mn, V, and Fe measured in sea water after seven days immersion were very low. This means that carbonated BOF slag produces less environmental burden.

Keywords: CO2 Capture, BOF Slags, Carbonization Process

1. INTRODUCTION

Blast furnace (BF) slag, basic oxygen furnace (BOF) slag, pre-treatment slag and secondary-refining slag are the four major types of slag that are produced in China Steel Corporation (CSC). Among these four slags, the amounts of BOF slag generated are surpassed only by that of BF slag. On average, 100 kg of BOF slag can be produced for each ton of crude steel manufactured, accounting annually for up to one million tons in CSC. The chemical composition of the BOF slag varies

depending on the raw material chemistry, steel grade produced and operating condition. Table 1 shows the main chemical compositions of BOF slag.

BOF slag has been used in various areas including road construction, cement making, soil treatment, industrial waste water treatment, precious slag balls manufacturing, wave dissipating concrete blocks, railway ballast material and in aquatic life preservation ^(3,4). Although BOF slag has found various applications, there are certain limitations which inhibit its complete utilization, especially for making powdered BOF slag (diameter, d_p

Component (wt%)	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	P ₂ O ₅	MnO	MgO	pН
China Steel Corporation	7-11	1.2-2.4	38-45	15-30	1.8-2.6	2.5-4.5	N.A.	12.1-12.4
Nippon Slag Association	11	1.9	45.8	17.4	1.7	5.3	6.5	N.A.
Indian condition	12-15	1-3	45-50	16-25	1-3.5	N.A.	1-2	N.A.

Table 1 Main Chemical Compositions of BOF Slag (1~3)

N.A .: not available

<4 mm). As shown in Table 1, BOF slag with CaO content up to 38-50 percent by weight (wt%) cause a high pH value and volume expansion resulting from the absorption of moisture or CO₂ by the free lime and magnesia leads to crack formation in the resulting structure. Carbonation is one of the methods used to reduce or eliminate the CaO content in the BOF slag. BOF slag must be weathered for 6-9 months or the free lime and MgO need to be stabilized before using it for any other purpose.

The reaction mechanisms, operating parameters and the characteristics of the BOF slag in the carbonation process have been investigated (3, 5-9). Mahoutian et al. investigated the carbonation and hydration behaviors of the BOF slag⁽³⁾. The results revealed that a longer carbonation time could promote the carbon dioxide uptake and strength gain. The compressive strength after 24 hours of carbonation was nearly twice as high as the strength after 2 hours of carbonation. Carbonation did not hinder the subsequent hydration performance of steel slag. The compressive strength achieved after carbonation and subsequent hydration was nearly close to the sum of carbonation strength and hydration strength. The strength gain in steel slag was dependent upon the content of calcium silicates in slag. Carbonation activation of calcium silicates of any polymorph could produce calcium-silicate hydrates intermingled with calcium carbonate crystals and increase the slag-bond strength. The steel slag containing high calcium silicates and low iron compounds would be ideal candidates to receive carbonation activation in its as-received form to make slag bond building products. Carbonation-activated slag products could be used to replace cement-based counterparts. Furthermore, as slag carbonation is a CO₂ uptake process, carbon dioxide could be sequestered through mineral precipitation of slag products to reduce CO₂ emission.

Pan et al. reported the accelerated carbonation of the BOF slag coupled with cold-rolling wastewater (CRW) in a rotating fixed bed (RPB) as a promising process for both CO2 fixation and wastewater treatment (5). Four particle size ranges including <125 µm, 125-350 µm, 350-500 µm, 500-840 µm, and 840-1190 µm were investigated. They founded that the CO₂-captured amounts increased with the decrease of particle size due to the fact that a smaller size of BOF slag caused more Ca²⁺ to dissolve. The results obtained from X-ray diffraction (XRD) spectra indicated that the major mineral phases reacting with CO₂ in BOF slag were Ca(OH)₂, Ca₂ (HSiO₄)(OH), CaSiO₃, and Ca₂Fe_{1.04}Al_{0.986}O₅. Meanwhile, the carbonation product was identified as calcite according based on scanning electron microscope (SEM), X-ray energy dispersive spectrometer (XEDS), and mapping results.

Chan compared the degree of aging of natural-aged

and rapid carbonated BOF slag from the physical and chemical properties of BOF slag ⁽⁶⁾. The results showed that the BOF slag with calcium content over 20 wt% had the potential to absorb CO₂. The calcium conversion rate of 6-month, naturally-aged BOF slag was nearly 15%. For rapid carbonation (24 hours) of BOF slag under the temperature of 100 °C, moisture content of 90% and 10 vol% partial pressure of CO₂, the conversion rate of BOF slag was upto 18%. Moreover, Chan also found that the rapid carbonation did not increase the leaching of heavy metals concentration, even under lowered pH environments (~11), BOF slag after a 24 hours rapid carbonation absorbed the amount of CO₂ up to 0.035 g-CO₂/g-BOF slag.

Nippon Steel Company has examined the fundamental experiments concerning about carbonation of steelmaking slag to solve the high pH water problem caused by alkaline contents from slag (7). Experimental results found that the slag carbonation reaction proceeded easily at room temperature under the optimized conditions for additional water content to the slag, relative humidity of atmosphere and flow rate of gas containing CO₂. It was confirmed that slag carbonation can be finished in a few hours even in the full-scale experiments in the works. In addition, Japan Fe Engineering (JFE) Steel developed a large steelmaking slag blocks by carbonation process of steelmaking slag (8). The slag materials were made by mixing 6 wt% of 0~5 mm BOF slag with water and carbonated under the high moisture, CO2 concentration of 25 vol%, and carbonation time of 72 hours. The density, pore ratio and compression strength of the carbonated aggregate were 2.2~2.4 g/cm³, 25-40%, and 100-190 kg/cm², respectively. It was conformed that these blocks were porous and the surface was covered with calcium carbonate. From the investigation on the possibility of using these blocks as a seaweed bed, these blocks were equal to concrete in the material characteristic and the hydraulic characteristic, and excellent in the algae creation effect. It was shown that the steelmaking slag blocks using carbonation process were useful materials for marine plant cultivation beds and artificial reefs from these results.

POSCO cooperated with Research Institute of Industrial Science and Technology (RIST) to restore the marine ecosystem with creating sea forests using steelmaking slags. They found that the steelmaking slag size of $20{\sim}40$ mm carbonated under the moisture content of 4.5% and CO₂ concentration of 25 vol% generated the sequestered amount of CO₂ of 0.3 ton-CO₂/ton-slag. In addition, the acceleration aging was applied to shorten the aging period. The immersion expansion ratio and the density of the aged steel slag were not more than 1.5% and not less than 1.5 ton/m^{3 (9)}.

There is potential for stabilized BOF slag by carbonation process generating a new environment-friendly steelmaking slag hydrated matrix used as a substitute for concrete blocks and semi-hard natural stones in various port construction works and seaweed forest to increase the application of BOF slag. However, the key factors affecting the properties of the carbonation performance and the relation between the carbonation parameters and the properties of carbonated BOF slag were not investigated thoroughly. In addition, the detailed carbonation technology of BOF slag was not easily obtained from Japan. Therefore, the objectives of this study were to install a lab-scale fixed bed carbonation system to investigate the key factors affecting the properties of carbonated aggregates and were familiar with the operating process of BOF slag carbonation.

2. EXPERIMENTAL METHOD

2.1 Installation of Carbonation System

Figure 1 shows a schematic of the carbonation system of BOF slag which included a gas supply, a fixed bed reactor, and a CO2 gas analyzer. The gas supply, containing 28-30 vol% CO₂ from the stack of a hot stove, was fed to the fixed bed reactor. The fixed bed reactor contained a column of BOF slag (dimension: 52 mm diameter and 150 mm height), a top cover, a bottom cover, and two diffusers at the inlet and outlet. The carbonation experiment of BOF slag included two steps: sample preparation and carbonation reaction. During the sample preparation, the BOF slag slurry was made by mixing 10-15wt% of powdered BOF slag with water. The slurry was then transferred to the fixed bed reactor to form an extruded column with an appropriate pore ratio. The carbonation reaction took place in the fixed bed reactor and the CO₂ concentrations at the inlet and outlet of the fixed bed reactor were measured with a CO₂ analyzer. The flow rate of the flue gas was controlled by a rotameter.

2.2 Characteristics Analysis of BOF Slag

The powdered BOF slag ($d_p < 4 \text{ mm}$) was provided

by CHC Corporation. Samples were dried at 105 °C for 1 hour in an oven prior to the experiments and then kept in a dryer. Parts of these samples were separated by shaking screen and weighed to measure the particle size distribution. In addition, the pH values of powdered BOF slag before and after the carbonation processes were measured by following Taiwan's National Institute of Environmental Analysis (NIEA) R208.04C method. The chemical composition of powdered BOF slag was measured by X-ray Fluorescence Spectrometer (XRF).

2.3 Dissolution Test of BOF slags

The pH values and metal dissolution of BOF slags were investigated in this study. The BOG slag and natural sea water were mixed to a weight ratio of 1:10 in an aquarium. The pH values of the sea water were measured by pH meter (Model pH 3110, Germany) each day of the immersion tests over a duration of seven days. The water samples were collected at the end of the dissolution tests and then measured for concentrations of chromium (Cr), manganese (Mn), vanadium (V), and iron (Fe) by inductively coupled plasma-optical emission spectrometer (ICP-OES) after sample filtration.

3. RESULTS AND DISCUSSION

3.1 Characteristics of Raw BOF Slag

3.1.1 Particle Size Distribution

Figure 2 shows the weight distribution of particle size of powdered BOF slag. The particle sizes more or less than 1.19 mm were about 55 wt% and 45 wt%, respectively. As for a particle size less than 1.19 mm, 15-16 percent by weight of the slag had particle sizes in the range of 0.59-1.19 mm, with 13-15 wt% with particle sizes of 0.25~0.59 mm, and 3-4 wt% with particle sizes with less than 0.074 mm. It revealed that the powdered BOF slag contained mostly small particles.

3.1.2 Chemical Composition

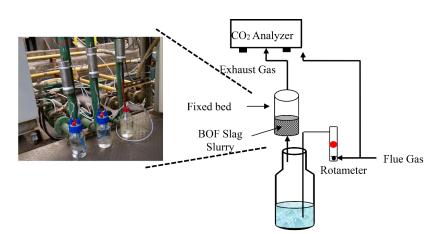


Fig.1. Schematic Diagram of CO₂ Capture and Carbonation System.

Figure 3 shows the chemical composition of various particle sizes of BOF slag using XRF. Each piece of data in Figure 3 is the average of three repetitions of each

sample measured. The main chemical composition of BOF slag consists of CaO (about 45-50 wt%), followed by Fe_2O_3 (approximately 24-32 wt%), SiO_2 (about 11-13

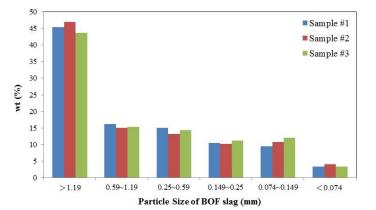


Fig.2. Weight Distribution of Particle Size of Powdered BOF Slag.

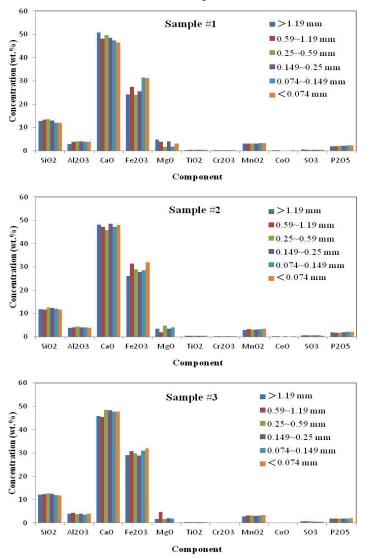


Fig.3. XRF Analysis of Powdered BOF Slag.

wt%), a little Al₂O₃, MgO, etc. In addition, the relations between the chemical composition and various particle sizes as well as the three samples were not evident. This data demonstrated that there were slight variations between various batches in the chemical composition of the powdered BOF slag.

3.1.3 pH Value

Any solution with a pH value higher than 12.5 or less than 2.0 is considered as hazardous industry waste according to the Taiwan's Environmental Protection Administration (EPA) law. The measured pH value of the powdered BOF slag was less than 12.5, and therefore the powdered BOF slag can be classified as a non-hazardous by-product of the steelmaking processes.

3.2 Characteristics of Carbonated BOF Slag

3.2.1 Appearance

The appearance of BOF slag changed from a slurry form to aggregates during the carbonation process. Before the sample preparation, the powdered BOF slag was mixed with water to form slurry. Equations 1 and 2 show calcium oxide within BOF slag dissolved in water and dissociated to aqueous Ca²⁺. Carbon dioxide in the flue gas flowed into fixed bed and dissociated to aqueous HCO₃⁻ and aqueous CO₃²⁻ while contacting with water, as shown in Equations 3 and 4. Calcium carbonate, the main carbonated product, was formed on the surface of BOF slag while aqueous Ca²⁺ reacted with aqueous CO_3^{2-} . The formed solid CaCO₃ acted as a cementing material, which made BOF slag into cylindrical aggregates with compression strength (Figure 4). There were some cavities and cracks that occurred on the surface of the right cylindrical aggregate. It was due to the sample preparation of the right cylindrical aggregate with a higher water loading ratio.



Fig.4. Appearance of Carbonated BOF Slag.

 $CaO_{(solid)}+H_2O_{(liquid)} \rightarrow Ca^{2+}_{(aqueous)}+2OH^{-}_{(aqueous)}.(1)$ $Ca_2SiO_{4(solid)}(larnite)+2H_2O_{(liquid)} \rightarrow$ $2Ca^{2+}_{(aqueous)}+H_2SiO_{4(solid)}+2OH^{-}_{(aqueous)}....(2)$ $CO_{2(gas)}+H_2O_{(aqueous)} \rightarrow H_2CO_3^{*}_{(aqueous)}....(3)$ $H_2CO_3^{*}_{(aqueous)} \rightarrow H^{+}_{(aqueous)}+HCO_3^{-}_{(aqueous)} \rightarrow$ $2H^{+}_{(aqueous)}+CO_3^{2-}_{(aqueous)} \rightarrow CaCO_{3(nuclei)}.....(5)$ $CaCO_{3(nuclei)} \rightarrow CaCO_{3(solid)}.....(6)$

3.2.2 pH Value

Table 2 shows the effect of the water loading ratio on the pH value of BOF slag aggregates. The pH values were measured following NIEA R208.04C. Apart from amount of water added, three carbonation tests were carried out under the same operating parameters, including CO₂ concentration, gas flow rate, carbonation time, and carbonation temperature. The amount of water added in the first, second and third tests were 15 wt%, 12 wt% and 10 wt% accordingly. The experimental results indicated that the pH value of the carbonated BOF slag were all less than 12.5. The more the amount of water added,

 Table 2
 Effect of Water Added on the pH Value of BOF Slag.

Samples	Ca	Raw Powdered BOF			
Operating parameters	#1	#2	#3	Slag	
Powdered BOF slag weight		500 g			
Amount of water added (water loading ratio)	75 g (15 wt%)	60 g (12 wt%)	50 g (10 wt%)	0 g (0 wt%)	
CO ₂ concentration		28 vol%			
Gas flow rate		3 L/min			
Carbonation time		72 hours			
Carbonation temperature	1	room temperature			
рН		<12.5		<12.5	

the lower the pH of the carbonated BOF slag was. As shown in Equations 1-6, water was an important reactant during the BOF slag carbonation process. Note that the water for the carbonation of BOF slag could be preloaded during the sample preparation, or injected through the diffusor during the reaction as steam. The lowest pH value of BOF slag in this study was near 12.

3.2.3 Immersion Expansion Ratio

The carbonated BOF slag was produced under the following operating parameters: water loading ratio of 15 wt%, CO₂ concentration of 28 vol%, gas flow rate of 3 L/min, room temperature, and carbonation time of 96 hours. In addition, the raw BOF slag was made into cylindrical aggregates by using under the pressure of 6,500 kg/cm². The immersion expansion ratios of BOF slag were measured by immersing samples in 80°C water and recording the length of the needle every day. Figure 5 shows the immersion expansion ratios of BOF slag before and after the carbonation process.

As shown in Figure 5, the raw BOF slag took 74 days to reach equilibrium and the immersion expansion ratios of samples #1 and #2 were 11.2% and 10.7%, respectively. The trends of the samples #1 and #2 were very similar and the variation of their data measured was due to the difference in the slag composition. In addition, the immersion expansion ratio increased fast in the beginning of 16 days and then slowed down until it reached equilibrium.

Moreover, the carbonated BOF slag took 37 days to reach equilibrium and the corresponding immersion expansion ratios were between 1.2-2.1%. The immersion expansion ratios of samples #1 and #2 were obviously higher than those of samples #3 due to the variation in the gas flow rate, the sample porosities, and the composition of the BOF slag. The time difference for these samples to reach equilibrium was due to their sample porosities. Despite the difference in the slag composition, the slope of the immersion expansion ratios for the carbonated BOF slag were all significantly lower compared to that of raw BOF slag. The overall results demonstrated that the carbonation process was effective in stabilizing the BOF slag.

3.2.4 Basic Physical Properties of Aggregates

Table 3 shows the effects of carbonation parameters on the compression strength of the carbonated BOF slag. The eight carbonation experiments in Table 3 were conducted at room temperature. Experimental results obtained from tests No.1-3 indicated that the carbonation time had a high impact on the compression strength of the cylindrical aggregates. The carbonation reaction started at the surface of BOF slag and then proceeded into the inner layers of the BOF slag. Therefore, the carbonation reaction of the BOF slag included two stages. The reaction rate in the first stage was rapid, since it was controlled by surface reaction. The reaction rate in the second stage was slow due to the fact that it was diffusion-controlled ⁽¹⁰⁾. The calcium carbonate formed fast on the surface of the BOF slag in the first stage, and it slowly accumulated on the pores of the cylindrical aggregates. The compression strength of cylindrical aggregates reached 34.58 kg/cm² after 24 hours of carbonation. However, the final compression strength increased only to 36.72 kg/cm² when the activation time was prolonged to 44 hours. Moreover, the gas flow rate was increased from 3 to 10 L/min in Tests No.2-3, but the compression strength only increased from 34.58 to 36.72 kg/cm² after an additional 20 hours of carbonation. The results suggested that the gas flow rate did not have a significant impact on the compression strength of the cylindrical aggregates. The

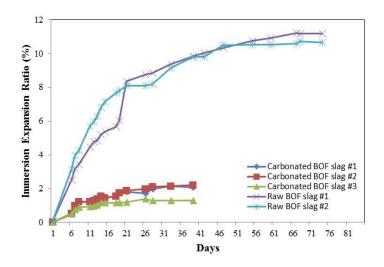


Fig.5. Comparison for Immersion Expansion Ratio of Raw and Carbonated BOF Slags.

No.		Operating Parameters				Basic Physical Properties of Aggregates			
	Water Added (wt%)	CO ₂ Conc. (vol%)	Gas Flow Rate (L/min)	Carbonation Time (hours)	Density (g/cm ³)	Pore Ratio (%)	Compression Strength (kg/cm ²)		
1	20	21	10	19	2.082	22.45	8.10		
2	20	20	3	24	2.008	20.83	34.58		
3	20	21	10	44	2.098	20.41	36.72		
4	10	28	2	48	1.933	23.08	9.40		
5	10	27	2	72	1.923	23.64	24.55		
6	10	28	3	72	1.956	21.95	11.82		
7	10	28	2	96	1.915	25.19	20.65		
8	10	28	3	96	1.907	24.07	22.20		

 Table 3
 Effects of Carbonation Parameters on Compression Strength of Carbonated BOF Slag.

results implied that the amount of CO_2 supplied was sufficient for the carbonation of the BOF slag at a gas flow rate of 3 L/min and CO_2 concentration of 21 vol%.

The results obtained from Tests No.3-5 demonstrated that water played an important role during the carbonation of BOF slag. As shown in Tests No.3-4, the water added was increased from 10 to 20 wt% while the compression strength obviously increased from 9.40 to 36.72 kg/cm². In addition, the water added was also increased from 10 to 20 wt% in Tests No.3 and 5, the compression strength increased from 24.55 to 36.72 kg/cm² even though the carbonation time shortened from 72 to 44 hours. More water was contributed to the dissolution of more Ca²⁺(aqueous) (Equations 1-2) to facilitate the carbonation reaction. Thus, it was necessary to prolong the carbonation time to increase the compression strength of the cylindrical aggregates while operating at a lower water loading ratio.

In addition, the results in Tests No.5-8 demonstrated that an extruded condition in the fixed bed was another factor affecting the compression strength of the aggregates. As shown in Tests No.5-6, the compression strength in No.5 was obviously higher than that in No.6 under the same operating parameters. Their variation between the compression strength of the carbonated BOF slag resulted in their different extruded condition. It can be approved from the difference of their pore ratios. In this study, the maximum achievable compression strength of carbonated BOF slag was 36.72 kg/cm² shown in No.3.

3.3 Dissolution Test of BOF slags

The experimental rig for the dissolution test of BOF slags are shown in Figure 6. An experimental group and a control group were included in this test. Three carbonated BOF slags, viewed as the experimental group, were produced under the following operating parameters: water loading ratio of 15 wt%, CO₂ concentration of 28 vol%, gas flow rate of 3 L/min, room temperature, and carbonation time of 72 hours. These carbonated BOF slags were assigned 0513CBOS, 0519CBOS, and 0526CBOS. Powdered BOF slag was viewed as the control group (Control BOS).

3.3.1 pH Value

Figure 7 shows the pH values of the sea water for the experimental group, the control group, and natural sea water. The results obviously indicated the pH values of the experimental group were lower than that of the control group, just slightly higher than that of sea water. It also shows the pH values of the experimental group can be controlled in the range of 8.38 and 8.84. However, the pH values of the control group suddenly increased to 9.5 in the first day, then slightly reduced to approximately 9 during $2\sim4$ days, and increased on pace to about 10. In addition, the pH values of the natural sea water varied in the range of 8.3 and 8.4.

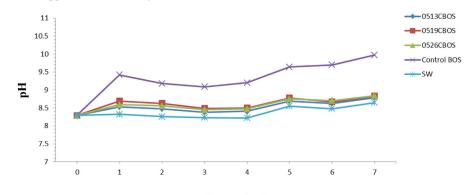
Moreover, no evidence of cavities or cracks occurred on the surface of the experimental group (Figure 8A) apart from some white crystallite (Figure 8B). In addition, it was monitored that huge amounts of white sediment were found in the control group (Figure 8C). The dissolution test demonstrated that the carbonation process was beneficial in reducing pH value of BOF slag.

3.3.2 Metal Dissolution

Table 4 shows the concentrations of four metal substances in sea water. The concentration measured in SW can be viewed as background concentrations. The analysis data shows that Cr concentrations of the experimental and control groups were slightly higher than that in natural sea water and were all under the standard value



Fig.6. Experimental apparatus of BOF slag immersed in sea water.



Immersion Days

Fig.7. pH values vs. immersion days for BOF slag and seas water

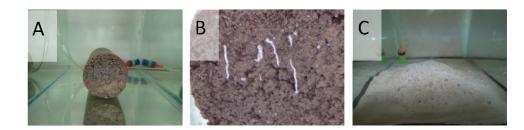


Fig.8. Surface conditions of carbonated BOF slag

Metal		Experimental Group	Control Group	SW	
	0513CBOS (mg/L)	0519CBOS (mg/L)	0526CBOS (mg/L)	(mg/L)	(mg/L)
Cr	0.002	0.003	0.003	0.007	ND
Mn	0.008	0.009	0.012	ND	0.001
V	0.001	0.001	0.004	ND	ND
Fe	0.001	ND	ND	ND	ND

ND: not detectable

of 0.05 mg/L referred to the Surface Water Classification and Water Quality Standards (SWCWQS) in Taiwan. The concentrations of Mn and V for the experimental group were slightly above those of the control group and sea water. The standard value of Mn in SWCWQS was 0.05 mg/L, while V was not included in SWCWQS. As for iron, the dissolution was almost negligible.

4. CONCLUSIONS

A lab-scale fixed bed carbonation system was installed in this study to investigate the properties of the carbonated BOF slag. The measurement results indicated that the powdered BOF slag contained up to 55 wt% of particle sizes less than 1.19 mm. The experimental results indicated that the appearance of BOF slag changed from a slurry form to aggregates with high compression strengths during the carbonation process. The experimental results indicated that the pH value of the carbonated BOF slag was less than 12.5. The increase in water loading lowered the pH values of the carbonated BOF slag. In addition, the immersion expansion ratio of the powdered BOF slag decreased from 10.1% to 2.1%. These results suggested that the carbonation process was beneficial in reducing the defects of the powdered BOF slag with high pH value and expansion ratio. Moreover, the experimental results indicated that the carbonation time had a high impact to the compression strength of the carbonated BOF slag. The amount of water added and the extruded condition in the sample preparation were another factors in increasing the compression strength of the carbonated BOF slag. The maximum achievable compression strength of the carbonated BOF slag in this study was approximately 36.72 kg/cm². Moreover, the results from the dissolution test of BOF slags indicated the pH of the sea water with the carbonated BOF slag was lower than that with the raw BOF slag, just slightly higher than that without BOF slag. The concentrations of Cr, Mn, V, and Fe measured in sea water after seven days immersion were very low. This means that carbonated BOF slag produces less environmental burden. This study revealed that it was valuable to further investigate into the application of using powdered BOF slag to capture the CO₂ emitted from flue gas.

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