Applicability of Carbonated Electric Arc Furnace Slag to Mortar

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Abstract. Authors have been studying the absorption of CO₂ in the steelmaking slag. In this study, an application of the electric arc furnace slag after the carbonation to admixture of mortar was investigated with the JIS (A6206-1997) method for ground granulated blast-furnace slag for concrete. The percent flows for the test mortar were smaller than that for the standard mortar. The percent flow of the carbonated slag whose average particle size of more than approximately 4 μm increased with an increase in the average size of the particles. Because the compressive strengths of the test mortar cured for 91 days were almost the same as those cured 28 days, the slag after the carbonation was thought not to have self-hardening property for a medium and long term. The compressive strength for the test mortar was almost unchanged within a range of approximately 2 to 7 μm of the average particle size, and it in this range was highest. The activity indexes for the test mortar prepared with the slag after the carbonation ranged from approximately 40 to 60 %.

1. Introduction

The increase in the atmospheric concentrations of the greenhouse gases has caused the global warming, which has brought about the climate change. The climate change will bring us into misfortunes, such as the catastrophe, the food shortages and ecocide, if present warming trends continue. Steel industries exhaust CO₂, which is one of the greenhouse gases, in reduction iron ores by cokes and operation. Meanwhile, they discharge a lot of slag. The slag contains CaO and MgO that react with CO₂. The behavior is discharged from the same place of emission source of CO₂. Then, we conceived adsorption and fixation of CO₂ in steel slag.

Until now, we have studied the absorption behaviors of CO₂ into a chemical reagent CaO, a waste concrete, metallic powders and slags that were discharged from electric arc furnace (EAF) under dry and wet grinding [1-6]. In the case that the EAF slags were wet ground in CO₂ atmosphere by a vibrating ball mill, most of the slags can absorb more CO₂ than the quantity of the emitted CO₂ that
was estimated from consumed electric power of the vibrating ball mill in operation. A large amount of the residue that is composed of precipitated CaCO$_3$ and finely ground unreacted slag is discharged from the process. A shortage of final disposal place in Japan limits the disposal of the slag on land. An ocean disposal of the slag is also considered, but the sea dumping does not readily advance even if safety of the slag is confirmed. From the viewpoint of effective use of resources, it is not good that the slag after the carbonation was put into the discard.

Blast furnace slag has been already used as cement and fine and coarse aggregates for concrete. Also, EAF oxidizing slag has been used as fine and coarse aggregates for concrete. Ground granulated blast furnace slag has also been used as an admixture for concrete and mortar. In addition to it, the fine inorganic particles, such as fly ash, silica fume, anhydrite and lime stone, have been used as admixtures to strengthen concrete and mortar. The usage of those slags, ashes and silica fume in concrete has been studied [7-26], and the usage of some of them is regulated by Japanese Industrial Standards (JIS) and so force. The fine particle whose size is less than about 5 μm is used as a repairing material of a crack of concrete. Some of the authors have studied on the application of inorganic materials, such as rock powders, sand and cement sludge, aluminum dross ash, oyster shell, glass fiber and fiber reinforced plastic to concrete [27-30].

The slag particles after carbonating treatment became small by wet grinding. Then, as a series of fundamental study of carbon capture storage utilized slag, the possibility of an application of the slag after the carbonation to admixture of mortar was investigated experimentally in this study to utilize them effectively.

2. Experiment

2.1. Carbonation of slag

The slags discharged from electric arc furnaces were used in this study. The chemical compositions of them were listed in table 1. The concentrations of metallic constituents were measured with an inductively-coupled plasma atomic emission spectrometry (ICP-AES) after the alkali fusion of the slag. Subsequently, the composition was calculated from the concentration obtained with an ICP-AES, assuming that the detected elements existed as compounds as shown in table 1. The slags discharged from EAFs are classified into two groups. One of them is oxidizing slag discharged after the smelting of oxidizing in an EAF. The slags OA, OB and OC are the oxidizing slag. The other is reducing slag discharged after the smelting process of reduction, and the slags RA and RB are the reducing slag. The slags OA, OB and RA, were discharged from an EAF in which stainless steel was smelted, and the others were discharged after normal steelmaking in an EAF. Generally, the reducing slag contains more CaO and CaS, and less MnO than the oxidizing slag. The normal steel oxidizing slag OC has more FeO than the stainless steel oxidizing slags, OA and OB.

The crystalline structure and the texture of slag are influenced by cooling condition. Figure 1 shows the X-ray diffraction (XRD) patterns of the slags as examples. All slags used in this experiment were not vitrified but crystallized. The detected crystals were also summarized in table 1.

Table 1. Chemical compositions and crystalline phases of used slag before carbonation

| Slag type | Chemical composition / mass% | Crystals |
|-----------|-------------------------------|----------|
|           | CaO  SiO$_2$  Al$_2$O$_3$  MgO  CaS  Cr$_2$O$_3$  MnO  TiO$_2$  FeO |          |
| OA        | 35.4  36.3  13.5  6.87  0.22  2.07  3.31  1.08  0.23 | 2CaO·MgO·SiO$_2$, 2CaO·Al$_2$O$_3$·SiO$_2$ |
| OB        | 33.0  44.1  5.39  7.68  0.33  3.29  4.09  1.02  0.74 | 2CaO·MgO·SiO$_2$, CaO·SiO$_2$·2CaO·Al$_2$O$_3$·SiO$_2$·Mg·Al(OH)$_3$ |
| OC        | 20.5  20.2  20.1  6.21  -  1.72  4.07  -  26.8 | 2CaO·Al$_2$O$_3$·SiO$_2$, 2CaO·SiO$_2$·FeO·Al$_2$O$_3$, FeO |
| RA        | 47.1  30.5  10.5  5.81  2.53  1.27  0.47  0.44  1.17 | 2CaO·SiO$_2$·MgO·SiO$_2$ |
| RB        | 50.3  21.1  18.7  4.12  4.02  0.19  0.60  -  0.93 | 2CaO·Al$_2$O$_3$·SiO$_2$, 2CaO·SiO$_2$·MgO |
Figure 1. XRD patterns of slag RA and OB. CaCO$_3$ (C) and (A) were the calcite and aragonite respectively.

Figure 2 shows the schematic of the experimental apparatus for carbonation [3, 4, 6]. This apparatus was used for measuring absorption of CO$_2$ with the constant pressure method. The slag of 0.4 kg in weight was set in the alumina lined grinding vessel. After the vacuum displacement of air in the container with CO$_2$, the purified water of 0.4 dm$^3$ in volume was poured into the container under CO$_2$ gas flow. Then, the slag was started the wet grinding using 180 alumina balls whose diameter was 20 mm. The pressure of CO$_2$ in the grinding vessel was kept to be 101.3 kPa by moving the piston of the gas volume meter.

![Apparatus used for carbonation.](image)

Figure 3 shows the behavior of CO$_2$ absorbed [3-6]. Except for the slags OA and OC, generally, the amount of CO$_2$ absorbed rapidly increased in the early stage of grinding, and, then, it gradually increased. As for the slags OA and OC, the amount of CO$_2$ absorbed increased almost linearly. In comparison of amount of CO$_2$ absorbed at grinding time of 14.4 ks, the amount of CO$_2$ absorbed in the
slag RA was highest, and those in the slags RB, OB, OA, OC decreased in this order. The slags used in this mortar test were wet ground for 14.4 ks. The slags OB, RA and RB whose amount of absorbed CO$_2$ became to be approximately half of that for 14.4 ks of grinding time were also used in this mortar test. The slag after wet grinding was passes through the filter whose pore was 0.45 μm in diameter. Then it was naturally dried. The slag hardened, but the agglomerated slag was so ductile that it was easily fractured by fingers. In addition, the agglomerate came apart apparently when it was dipped into water. The slag agglomerate was sieved, and it whose diameter was less than 850 μm was used for the mortar test.

Table 2 shows the specimens used for the mortar test. The specimen OB0 was unground slag. Figure 1 shows the XRD patterns of the specimen as examples. CaCO$_3$ was detected in the wet ground slag. The intensity of the peaks for the CaCO$_3$ increased with an increase in the grinding time. Conversely, the intensities of the peaks for the other substances that were originally contained in the slag decreased or disappeared as the grinding progressed. The crystalline phases of the slags after the carbonation were summarized in Table 2. The CaCO$_3$ was estimated to form by the reaction between Ca$^{2+}$ eluted from the slag and CO$_3^{2-}$ dissolved from CO$_2$ occurred at the interface between water and CO$_2$. The overall formation reaction of it was expressed by:

$$\text{CaO + CO}_2 = \text{CaCO}_3$$

Based on eq. (1), the carbonation ratio, $CR$, in table 2 was calculated by:

$$CR = \frac{Q_{\text{CO}_2}}{N_{\text{CaO}}} \times 100$$

where $Q_{\text{CO}_2}$ is the absorbed CO$_2$ [mol], and $N_{\text{CaO}}$ is the amount of substance of CaO in slag [mol]. In our previous work [6], the relation between the pH and the concentration of Mg dissolved in water during grinding suggested the formation of MgCO$_3$·3H$_2$O that was not detected by a XRD. Because the quantity of formation of it could not be evaluated, the carbonation ratio was calculated assuming that the CO$_2$ absorbed reacted with only the CaO in the slag. In addition, the relation between the pH and the concentration of Ca dissolved in water suggested the formation of CaCO$_3$·6H$_2$O. However, the formation of it does not influence the carbonation ratio. Figure 4 shows the specimens after the carbonation. Those particles were plate like.

According to the standard, the specific surface area of the slag particles should be measured with the permeation method. As mentioned before, the slag after carbonation cohered. The gas permeation method like the Blaine method does not give accurate specific surface area of the agglomerated particle. Then, size of the slag particle was measured from the image as shown in figure 4. Table 2 shows the range and average of the particles size. The average size of particle ground for short time was larger than that for long time. The average size of the specimen OC4 was larger than that of the other ground specimens. This thing resulted from that the slag OC was difficult to be broken because the slag OC contained much FeO in itself.

**Figure 3.** Behavior of absorbed CO$_2$ in each slag.

**Figure 4.** Carbonation treated slag particles for 14.4 ks.
Table 2. Slag used for preparation of mortar.

| Specimen | Slag | Grinding time /ks | Carbonation ratio, CR-% | Particle size /μm | Crystals |
|----------|------|-------------------|------------------------|-------------------|----------|
|          |      |                   |                        | Range             |          |
|          |      |                   |                        | Average           |          |
| OA4      | OA   | 14.4              | 24                     | 1-15              | 4.4      |
| OB0      | OB   | 0.0               | 0                      | 100-850           | 450      |
| OB2      | OB   | 7.2               | 41                     | 1-10              | 5.1      |
| OB4      | OB   | 14.4              | 80                     | 0.5-7             | 2.3      |
| OC4      | OC   | 14.4              | 21                     | 2-40              | 14.1     |
| RA1      | RA   | 3.6               | 50                     | 1-10              | 6.5      |
| RA4      | RA   | 14.4              | 85                     | 0.5-8             | 3.5      |
| RB1      | RB   | 3.6               | 49                     | 0.5-6             | 4.0      |
| RB4      | RB   | 14.4              | 71                     | 0.5-5             | 1.8      |

2.2. Flow and compressive tests

The preparation of mortar, the flow test and the compressive test was performed based on the JIS A6206-1997 (Ground granulated blast-furnace slag for concrete). This regulation contains some of the matter of the JIS R 5201-1997 (Physical testing method for cement). Here, those are explained briefly.

The standard mortar was prepared with 0.450 ± 0.002 kg of the ordinary portland cement, 1.350 ± 0.005 kg of the standard sand that was natural silica sand whose size distribution was adjusted, and 0.225 ± 0.001 kg of the tap water. The test mortar was prepared with displacing half of the cement to the carbonated slag. Therefore, it was prepared with 0.225 ± 0.002 kg of the portland cement, 0.225 ± 0.002 kg of the slag, 1.350 ± 0.005 kg of the standard sand and 0.225 ± 0.001 kg of the water. These materials were kneaded by the mixer respectively. After that, the mortar was poured in the flow cone. After the flow cone was removed rightly above, it was vibrated fifteen times for fifteen seconds. The flow value was obtained from averaging the maximum diameter of the cone and the diameter that was in the orthogonal direction of the maximum diameter.

The kneaded mortar was also poured in the metallic die. The shape of the mold mortar was a square prism (40×40×160 mm). The mortar was set in the box in which the relative humid and the temperature were controlled to be over 90 % and 293 ± 1 K respectively. Subsequently, it was hold for twenty seven or ninety days in the water whose temperature was 293 ± 1 K. The compressive strength was measured by the compression testing machine (Shimadzu UMH 100). The test piece for measurement of the compressive strength was prepared by cutting the hardened mortar by half. Three of them were used for the compressive test.

3. Results and Discussions

3.1. Flow value

The flow value represents the fluidity and the softness of mortar. Figure 5 shows the percent flow of each specimen. Here, the percent flow, $F [%]$, is the ratio of flow values, and it is defined by:

$$F = \frac{l_2}{l_1} \times 100$$

where $l_1$ and $l_2$ are the flow values of the standard and the test mortar respectively. The flow value of the standard mortar was 188. The percent flows of the specimen OB4, RA4 and RB4 could not be
measured, because their mortar cracked under the vibration. Generally, the percent flows of the test mortar were lower than that of the standard mortar. It was reported that the flow value for the mortar blended with fine ground particles of the molten slag that was discharged from a garbage incineration plant was almost the same as the standard value [17]. The percent flow of the carbonation treated slag seemed to be inferior to that for the molten slag. In the case that the finely ground mineral powder used in concrete paste, the more amount of water has to be added into paste to enhance and hold the workability [31]. Therefore, addition of much water or an air–entraining or a water reducing admixture and so on is thought to be necessary to improve the flow value of the test mortar.

The percent flows of the specimen of OB0, OB2 and OB4 decreased in this order. Because the carbonation ratios of OB0, and OB2 and OB4 increased in this order, the percent flow decreased with an increase in the carbonation ratio. The low carbonation ratio means the large quantity ratio of the unreacted CaO to the total CaO in slag. The unreacted CaO is thought to contribute to the hardening of the mortar, and the percent flow is estimated to be lowered by the hardening. However, the percent flow of the mortar that contained much unreacted CaO was larger than that contained less unreacted CaO. This is thought to mean the percent flow is not necessarily influenced by the chemical property of the slag.

As for the fluidity of the cement paste that was mixed with fine limestone, it was reported that there was the optimal conditions of the combination of the roundness and the distribution of the particles [32]. Then, it was investigated that the effect of the average size of the slag on the percent flow, and the relation of both was shown in figure 6. As mentioned above, the flow values of the specimen OB4, RA4 and RB4 could not be measured. The average particle sizes of them were below approximately 4 μm. The free water is well-known to contribute the fluidity [31, 32]. These smaller particles hold water in the space among them, and the particles cohered each other [33]. This results in a decrease in the quantity of the free water, and seemed to lower the ratio of flow values. Except for percent flow of the unground specimen OB0, the percent flow for the average size of more than approximately 4 μm showed the upward tendency with an increase in the average size, but the tendency was not clear. Probably, the shape and distribution is thought to affect the ratio of flow value.

Figure 5. Percent flow of each test mortar.

Figure 6. Relation between percent flow and average particle size of slag after carbonation.

3.2. Compressive strength
Whereas the flow values of the specimen OB4, RA4 and RB4 could not be measured, it was possible to put them in the mold and make their test pieces for the compressive test. The amplitude and frequency for making the test piece were 0.8 mm and 2800 Hz, and those for measuring the flow value were 10 mm and 1 Hz. The small amplitude and large frequency seemed to make it possible to fill their mortar in the mold well, and cracking of their compacts was thought not to occur at the time of separation of them from the die.

Figure 7 shows the compressive strength of each mortar. The compressive strengths of the test mortars were smaller that of the standard mortar. The compressive strength of the mortar blended
with fine molten slag particles decreased with an increase in the replacement ratio of the slag to cement [17]. The compressive strength of the mortar in which the replacement ratio was 25% was almost the same as that of the standard mortar in which the ratio was 0%, but it for 50% of the ratio became smaller than that the standard [25]. In this work, the replacement ratio was 50%. The smaller compressive strengths for the test mortars seemed to be similar to those for the mortar mixed with the molten slag.

As shown in Figure 7, the compressive strengths of the test mortar cured for 91 days were larger than that for 28 days. However, because the 95% confidence limit of the measured compressive strength was ±3 MPa, it may be said that only the difference in the compressive strengths of the specimen OB0 was the meaningful. Therefore, the slag OB, which was not carbonated, has self-hardening property, whereas the slags after carbonation seem not to have self-hardening property in the term of 28 to 91 days.

The compressive strengths for the specimens OB2 and OB4 were almost the same, and those were larger than that for the specimen OB0. The compressive strengths for the specimens RA1 and RA4 were almost the same, whereas the compressive strength for RB1 was larger than that for RB4. The carbonation ratios of the specimens OB0, OB2 and OB4 increased in this order. Similarly, the carbonation ratio of the specimen RA1 and RA4, RB1 and RB4 increased in this order respectively. Therefore, the compressive strength for the test mortar was not influenced by the carbonation ratio. The low carbonation ratio means that the much unreacted CaO exists in the slag. This means that the unreacted CaO does not contribute to the hardening of mortar.

As shown in figure 5, the percent flows of the specimens OB4, RA4 and RB4 could not be measured. The compressive strength of the specimens OB4 and RA4 were relatively large, those were almost the same as the specimens OA4, OB2, RA1 and RB1 whose percent flows over approximately 60%. Accordingly, the percent flow did not influence the compressive strength.

Figure 8 shows the relation between the compressive strength and the average size of the slag. The compressive strength was almost constant over a range of approximately 2 to 7 \( \mu \)m of the average particle size. When the average particle size exceeded about 7 \( \mu \)m, it decreased with an increase in the average size. This result corresponds with the results of the molten slag [17]. The compressive strength for about 1 \( \mu \)m of the average size was small. This seems to result from cohesion of the slag particles [33].

The compressive strengths of these test mortar are lower than those of mortar for concrete, but there are mortar that have such strengths. Fine particles whose average size are a few micron meters have been used as injectable materials to grout cracks. These test mortar seem to be able to be used such repairing materials.

In the JIS, the compressive strength of mortar is evaluated by the activity index. Here, the activity index, \( A_s \), is defined by

\[
A_s = \frac{c_2}{c_1} \times 100
\]

where \( c_1 \) and \( c_2 \) are the compressive strengths of the test pieces of the standard and test mortar cured for the same time respectively. Figure 9 shows the activity index of each specimen. Because the compressive strength of the standard mortar cured for 91 days was almost the same as that for 28 days, the relation between the activity index and the test mortar was similar to that between the compressive strength and the test mortar as shown in figure 7. The activity index of the test mortar was within a range of approximately 40 to 60% except for the specimen OB0 which was unground slag. The activity indexes for the test mortar were smaller than those regulated by the JIS A 6206 and the ASTM C 989 for the ground granulated blast–furnace slag. The slag after carbonation was not used as alternate material of the ground granulated blast–furnace slag.
4. Conclusions

In this study, it was tried that the EAF slag after carbonating treatment was applied to the admixture of mortar. A flow value and a compressive strength of test mortar were measured based on the JIS A6206-1997 (Ground granulated blast-furnace slag for concrete). The obtained results could be summarized as follows:

1. The percent flows for the test mortar were smaller than that for the standard mortar. The average particle size of the carbonated slag influenced the percent flow. The percent flows for the average particle size of less than approximately 4 μm could not be measured. The percent flows for the carbonated slags whose size were larger than approximately 4 μm increased with an increase in the average size of the particles.

2. It was possible to make the compact for compressive test with mortar whose flow value could not be measured.

3. The compressive strengths of the test mortar cured for 91 days were almost the same as those cured 28 days. It was thought from this result that the slag after the carbonation did not have self-hardening property for a medium and long term.

4. The compressive strengths for the test mortar were smaller than that for the standard mortar. The activity indexes for the test mortar prepared with the slag after the carbonation ranged from approximately 40 to 60%.

5. The compressive strength became large when the average particle size became large from approximately 1 to 2 μm. The compressive strength for the test mortar was almost unchanged within a range of approximately 2 to 7 μm of the average particle size. When the average particle size exceeded about 7 μm, the compressive strength decreased with an increase in the average size.
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