Construction and Building Materials 107 (2016) 191–202

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Autogenous and engineered healing mechanisms of carbonated steel slag aggregate in concrete

Bo Pang, Zonghui Zhou *, Pengkun Hou, Peng Du, Lina Zhang, Hongxin Xu

Shandong Provincial Key Laboratory of Preparation and Measurement of Building Materials, Engineering Center of Advanced Building Materials of Ministry of Education, University of Jinan, Jinan 250022, China

HIGHLIGHTS

- CSA is used as autogenous healing material in concrete.
- The investigation on repeated fracturing and healing.
- The composition and morphology of healing products are investigated.
- The ultrasonic test was used to analyze the cracks and healing condition of specimens.

ARTICLE INFO

Article history:
Received 4 September 2015
Received in revised form 25 December 2015
Accepted 26 December 2015
Available online 12 January 2016

Keywords:
Carbonated steel slag
Aggregate
Concrete
Self-healing

ABSTRACT

The application of self-healing technology in concrete materials was widely investigated in the past decade. Although the micro-capsules and bacteria were considered as promising self-healing agents to realize durability enhancement of concrete, the high cost of micro-capsules and limited bacteria types are still big challenges that limit the widely application of this technology. As a result, it is necessary to develop cost-effective and environmentally friendly materials as self-healing agent in concrete. In this study, carbonated steel slag was used as aggregate to realize the autogenous healing of concrete. The self-healing performance of this concrete was investigated by comparing with the concretes prepared with normal aggregates and crushed steel slag aggregates. In addition, the hydration heat, X-ray diffraction, and scanning electron microscopy/energy dispersive spectroscopy results were analyzed to elucidate the self-healing mechanisms of concrete via using carbonated steel slag as healing agent. It was found that the healing products are mainly composed of $CaCO_3$, $Ca(OH)_2$, calcium–silicate–hydrate, calcium–aluminate–ferrite hydrate as well as amorphous silica. The cracks of aggregate have been healed to a certain extent that maximum healing width is about 20 μm and maximum healing length is about 5 mm.

1. Introduction

Portland cement is one of the most widely used binder materials to prepare concrete. The global cement production reached 4.6 billion tons in 2014 [1]. However, the production of cement is an energy-intensive process that significantly contributes the emissions of greenhouse gases. In addition, the average service lives of many concrete constructions are not as long as they were designed when they are exposed to extreme service conditions [2–4]. As a result, the budgets on reconstruction and maintenance activities of concrete constructions keep increasing every year [5,6]. In order to reduce the costs and mitigate the negative environmental footprints of reconstruction and maintenance activities of concrete structures, self-healing technology was investigated in concrete to enhance its durability [7–9].

The self-healing concretes were divided into two main streams according to the healing mechanisms, namely the autogenous healing and the engineered healing [10–14]. The engineered healing is realized by adding specific chemical or biological agents into the cementitious matrix, while the autogenous healing is resulted from the production of healing contents through the internal chemical reactions of the cementitious matrix itself [8,10,11,15].

The engineered healing agents can be divided into several categories: hollow fibers or micro-capsules, bacteria and shape memory materials [16]. The healing mechanism of the engineered healing is based on the release of the healing agents, which were contained in hollow fibers or micro-capsules, when cracks are initiated in concrete. Dong et al. [17] invented a self-healing system

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* Corresponding author.
E-mail addresses: pbo1990@hotmail.com (B. Pang), mse_Zhouzh@ujn.edu.cn (Z. Zhou), xuhx1990@hotmail.com (H. Xu).

http://dx.doi.org/10.1016/j.conbuildmat.2015.12.191
0950-0618/

which can release the healing agent and activate the healing effects when cracks are generated in cementitious composites. Hilloulin et al. [7] presented the design of polymeric capsules which are stable during the mixing process but will break when cracks initiate.

In spite of the benefits of using hollow fibers or the micro-capsules as healing agent is undeniable, the weakness of them is still distinctive. They will become internal defects or stress concentration points after the healing agents were released. Moreover, the hollow fibers and micro-capsules are one-time materials as healing agents along with relatively high cost, therefore, their application in self-healing concrete has been largely limited.

Apart from the hollow fibers or micro-capsules, a few types of bacteria were used as self-healing agents to prepared engineered healing concrete. Although the cultivation and isolation of bacteria are not challenging technologies so far, the types of bacteria which can stably survive in concrete environment, such as high temperature and high pH value, are still very limited. Therefore, it is necessary to develop cost-effective and environmentally friendly materials as self-healing agent in concrete materials.

Steel slag is a by-product during the steel manufacturing process. The recycling use of the steel slag can largely mitigate the solid waste disposal problems [18–20]. Carbonation of steel slag is an effective way to stabilize its active composition, such as free CaO and MgO, which considerably expanded its application as aggregates in concrete production. Other than mitigating the solid waste disposal problems, the carbonation process of the steel slag is a potential method to simultaneously capture and store CO_2 from point-source emissions [21–23]. Although using steel slag as aggregates in concrete has been widely investigated for many years [24–26], the research on the application of carbonated steel slag (CSA) as aggregates to realize the self-healing concrete, to the best of our knowledge, is still in its infant stage. In this context, this work is devoted to investigate the self-healing effects of CSA used as aggregate in concrete, and elucidate the healing mechanisms of this autogenous healing concrete.

1.1. Cost of CSA

The cost of steel slag is 9.372 USD/t, and the cost of CO_2 (99.5% of purity) is 0.62 USD/kg. In the previous study, the average amount of carbon dioxide solidification is about 5–8 wt.% [25]. Thus, the material cost of manufacturing aggregate is about 9.66 USD/t. However, if carbonation devices are built around the cement or iron and steel plants, while the industrial waste gas and waste heat are reused to produce CSA, the costs will be further reduced.

2. Experimental

2.1. Materials

Considering the main components of BOF slag are calcium silicate and calcium ferrite which provide CSA the potential hydraulicity and cementitious properties, the BOF was used to manufacture the aggregate. When the CSA gets cracked, the internal clinker minerals can react with water inside to trigger the healing effect. Portland cement with the Blaine fineness of 311 m^2/kg (according to the ASTM C150/C150M-12 [27]) was used to prepare the concrete samples. The Basic Oxygen Furnace (BOF) slag provided by Jinan Iron and Steel Works Company (ground by a lab ball mill for 30 min and then sieved through a sieve of 600 μm) was used as raw materials for preparing CSA. The chemical composition of the cement and steel slag are listed in Table 1 and the particle size distribution is shown in Fig. 1.

The concretes with normal aggregate (NA) and crushed steel slag (SSA) were prepared for comparison. For the NA concrete samples, the coarse aggregate were crushed limestone with size of 4.75–18 mm and the fine aggregate was natural river sand with size of 0.075–4.75 mm. For the SSA samples, the coarse and fine aggregates were all crushed raw steel slag with the size distribution as same as the NA.

2.2. Preparation of CSA

2.2.1. Pelletization

The water was sprayed on the steel slag powder mixture by pelletizer. The formation of pellets occurred between 10 and 12 min in trial productions. The total pelletization time was determined as 20 min for the compaction of fresh pellets.

2.2.2. Carbonation

The untreated CSA was carbonated in a carbonation chamber. The chamber was heated up to 70 °C and vacuumed to −0.03 MPa before carbonation. Then CO_2 was introduced into the reactor until the pressure reached 0.3 MPa. The carbonation time was 4 h and the technological process is shown in Fig. 2.

2.3. Concrete samples preparation

The Fuller distribution was applied to amend the grading curve of the aggregates. Considering shape effects which can affect the bulk density between the CSA and the other two aggregates, correction factors of 0.33 [28] and 0.5 [29] were applied (the distribution of aggregate is shown in Fig. 3). The concrete was prepared with a W/C ratio (Water to cement ratio) of 0.5 and cured at the RH of 95% and the temperature of 20 ± 2 °C. The cement content per cubic meter was 375 kg. Cubes (100 mm^3) were cast and tested according to ASTM C39M [30].

2.4. Healing property evaluation

2.4.1. Pre-cracking process

The 28 days cubic concrete samples with size of $100 \times 100 \times 100$ mm^3 were used to obtain the pre-cracking samples. The maximum loading applied on the fresh concrete was set as the first crack value. Once the loading reached this value, it was automatically stopped, and the pre-cracked samples were moved to the curing room (95–98% RH and 20 ± 2 °C). The autogenous healing process was lasted for 3 months. The detailed recurrent fracture program is shown in Fig. 2(d).

For a better understanding about the healing behavior of CSA in concrete, several randomly selected CSAs were broken into halves and pieced together (shown in Fig. 2c). They were then divided into two groups, one group was stored in water and the other one was stored in curing room. The healing states of such CSAs were observed under microscope after seven days, one month and three months, respectively.

2.4.2. Ultrasonic testing

The ultrasonic signal was generated and transmitted by Tektronix AFG3022B signal generator while received and displayed by Tektronix TDS1002B-SC oscilloscope. The frequency of the ultrasonic was 50 kHz. The amplitude, sound velocity and frequency of ultrasonic wave were collected and recorded by PROCEQ TICO ultrasonic detector. The generator and the receiver were placed vertically on both sides of the cubic concrete samples with foam pad cushioned under the samples and Vaseline as a coupling agent (shown in Fig. 2b).

2.4.3. Strength recovery after recurrent fracture program

In order to investigate the strength recovery of the samples after recurrent fracture, the cracked and cured samples were re-cracked to the maximum load and then cured for further three months (logic diagram of the recurrent fracture program is shown in Fig. 2d). One pre-cracked specimen in each proportion (without healing treatment) was re-cracked three months later, and its stress–strain curves were used as the control curve. The recurrent fracture program was repeated until the tested strength was basically coincided with that of control curves.

The tests are performed in displacement control. The maximum displacement is about 0.25 mm and the width of apparent cracks is less than 0.3 mm. In order to prevent the failure of the samples, when the loading reached up to ultimate strength before displacement up to 0.25 mm, the loading will be stopped. In the 2nd, 3rd and 4th fracture treatment, when the widths of apparent cracks are larger than 0.5 mm, the loading will be stopped immediately.

2.5. Hydration heat

The hydration heat evolution rate and cumulative hydration heat of CSA and cement paste aged for 28 days were measured with an isothermal calorimeter (TAM Air from TA instruments). Tests were performed at water to binder ratio (W/B) of 0.5 within 7 days.

2.6. Microstructure analysis

The healing condition of cracked CSA was observed in the fracture area under microscope with curing age of 7 days, 1 month and 3 months respectively. In order to better observe the healing phenomenon at the seam spots of aggregates, the aggregates were polished at the place vertical to the seams. Furthermore, the