Internal curing effect of raw and carbonated recycled aggregate on the properties of high-strength slag-cement mortar

Selamu Yihune Abate\textsuperscript{a}, Keum-Il Song\textsuperscript{b}, Jin-Kyu Song\textsuperscript{b}, Bang Yeon Lee\textsuperscript{b}, Hyeong-Ki Kim\textsuperscript{a,*}

\textsuperscript{a}School of Architecture, Chosun University, 309 Pilmun-daero, Dong-gu, Gwangju 61452, Republic of Korea
\textsuperscript{b}School of Architecture, Chonnam National University, 77 Yongbong-ro, Buk-gu, Gwangju 61186, Republic of Korea

\textbf{HIGHLIGHTS}

- RRA and CRA were used in high-strength slag-cement mortar for internal curing.
- Incorporation of RA reduced both autogenous and dry shrinkage of the mortar.
- Internal curing efficiency of recycled aggregates was improved by their carbonation.

\textbf{ABSTRACT}

The internal curing effect of raw and carbonated recycled aggregates on the properties of high-strength slag-cement mortar was investigated. First, the aggregates were characterized using different methods and their effects on the shrinkage, drying mass loss, non-evaporable water content, and compressive strength of the mortar were evaluated. Their incorporation reduced the shrinkage and improves non-evaporable water content, i.e., degree of hydration, with a slight reduction in compressive strength. Because of the carbonation of the aggregates, the mixtures incorporating carbonated aggregates show lesser improvements in non-evaporable water content and compressive strength than the ones with non-carbonated aggregates. However, the shrinkage property was better improved by the incorporation of the carbonated ones.

\section{1. Introduction}

The production of aggregates from hardened concrete is a critical operation that contributes not only to solve the growing waste disposal problem but also assists in conserving natural resources like sand and gravel and securing the future supply of materials for construction \[1\]. In locations where good-quality aggregates are scarce and the cost of disposal of concrete rubble is high, the use of concrete rubble as a source of aggregate is a feasible operation that has been used in recent decades \[2\]. Although the cost of crushing, grading, dust control, and separation of undesirable constituents is regarded as an obstacle, the production of recycled aggregates by crushing of concrete pavements and massive structures has proved to be an economical source of aggregate to produce new concrete \[3\].

According to ASTM C 294 \[4\], the recycled aggregates recovered from hardened concrete are classified as an artificial aggregate. EN 13055-1 \[5\] specifies the properties of lightweight aggregates (including recycled aggregates) for use in different types of construction. Depending on their quality, recycled aggregates sourced from construction and demolition operations can be employed in roads, drainage, and other construction projects, including structural concrete production \[6\]. A substantial number of studies have reported different properties of recycled aggregates and the concrete containing them. As these studies indicate, the amount of attached mortar, strength of concrete to be recycled, composition of recycled aggregate, and many other factors have a critical influence on the properties of recycled aggregates and on the concrete containing them \[7–10\].

If the recycled aggregates are well saturated prior to mixing, the higher water absorption properties of these aggregates could be used as internal curing agents \[10\]. Internal water-curing methods using pre-saturated aggregates are not only beneficial to promote hydration, but also to minimize shrinkage and compensate for
the evaporation loss of water from the concrete [11,12]. In some cases, the incorporation of an internal curing agent enhances the durability of the concrete in many ways, such as increasing the degree of hydration; reducing the porosity, reducing internal stresses, and cracking owing to shrinkage; and causing densification of the interfacial transition zone [13]. Considering the mechanical properties, the introduction of internal curing agents can have an influence on the performance, i.e., increasing the amount of curing agent might cause a reduction in strength [12].

Cusson and Hoogeveen [14] examined the effectiveness of pre-soaked fine lightweight aggregates as an internal curing agent to reduce the autogenous shrinkage of high-performance concrete. In this study, the mixture with an adequate amount of internal curing agent showed a high internal relative humidity after seven days and eliminated the autogenous shrinkage completely without affecting the strength and stiffness of the concrete (measured after seven days). Moreover, it eliminated the tensile stress owing to the net autogenous shrinkage. Zhutovsky et al. [15] also studied the influence of internal curing on cement matrix of high-performance concrete with different water-to-binder ratios (w/b) and levels of internal curing replacement. In this study, the reduction in w/b resulted in a slight reduction on internal curing efficiency; however, the addition of the silica fume in the mixture resulted in a drastic reduction on internal curing efficiency. This was attributed to the greater tightness of the silica fume mix, resulting in a short distance over which water could exit from the lightweight aggregate to penetrate the surrounding matrix. Zhutovsky and Kolver [16] studied the effect of internal curing on durability-related properties of high-performance concrete. The chloride penetration varied with w/b; an improvement was observed for a w/b of 0.33, but the effect was not significant at lower w/b ratios. For the dry shrinkage test, the lower w/b showed greater improvement than the higher values. In general, the inclusion of internal curing agents caused no serious degradation in durability-related properties. Sagoe-Crentsil et al. [17] reported that, for similar volumetric mixture proportions, the mixture containing recycled aggregates and conventional aggregates (basalt) exhibited similar mechanical strength. However, the dry shrinkage of the recycled aggregate concrete had higher values than the control mixture. Which is attributed to the lower restraining capacity of recycled aggregates than the basalt aggregate. Kim and Bentz [18] evaluated the feasibility of recycled fine aggregate as an internal curing aggregate for high-performance concrete. In this study, crushed returned fine aggregate, i.e., artificial recycled aggregates crushed from newly manufactured concrete, was used as a substitute for saturated lightweight aggregate. The sole use of the aggregates had a limited influence on the reduction of autogenous shrinkage, and also yielded a significant reduction in strength. However, the autogenous shrinkage of the concrete was mitigated significantly without any reduction in strength by using a combination of crushed returned aggregates with artificial lightweight aggregates.

Recycled aggregate produced under actual conditions may have different internal curing efficiencies under the carbonation state. As unhydrated cement remained on the surface, the recycled aggregate is classified as a kind of reactive aggregate. However, according to previous research results [19,20], when the surface of the internal curing aggregate is reactive, the surface pores may be clogged and, since these are the pathways of moisture transfer, the shrinkage reduction efficiency may decrease. Meanwhile, if the recycled aggregate is carbonated because of various factors, such problems may not occur.

This experimental investigation aims to evaluate the influence of incorporating raw and carbonated recycled aggregates in high-strength slag-cement mortar as internal curing agents. In addition to the control mortar mixture, a mixture with expanded shale, i.e., artificial lightweight aggregates, was prepared as a reference. The aggregates were characterized using X-ray diffraction (XRD), thermogravimetric analysis (TGA), mercury intrusion porosimetry (MIP), ex-situ leaching pH measurement, and phenolphthalein spraying. The effects of recycled aggregates were evaluated in terms of compressive strength, non-evaporable water content, autogenous shrinkage, dry shrinkage, and drying mass loss of the mortar. Note that the non-evaporable water content is an indirect indicator of the degree of hydration of the mixtures, which is used for evaluation.

2. Materials and methods

2.1. Materials

2.1.1. Binder

Type I Ordinary Portland cement (OPC) in accordance with ASTM C150 [21] and blast furnace slag (BFS) satisfying both the Korean Standard (KS) F 2563 Type 3 [22] and ASTM C 989 Grade 100 [23] were used as binders for all prepared mixtures. The OPC has a specific gravity and blain fineness of 3.15 and 3495 cm²/g, respectively. Whereas for the BFS, the specific gravity and blain fineness is 2.90 and 4215 cm²/g, respectively. The chemical composition of the binders are presented in Table 1.

2.1.2. Aggregates

The recycled aggregate used in this experimental program was produced by a local construction waste disposal facility (Gwangu, Republic of Korea). The aggregates that passed through a 2-mm sieve and the aggregates that retained on 12.5 mm sieve were discarded. Then, some portion of the recycled aggregates were subjected to an accelerated carbonation treatment in a chamber with 5% CO₂ at 20 °C and 50% relative humidity for seven days. Therefore, the recycled aggregates before and after the carbonation treatment are designated as a raw recycled aggregate (RRA) and carbonated recycled aggregate (CRA), respectively. In addition, the lightweight aggregate, expanded shale (ES), was also used as a reference internal curing aggregate. For all prepared mixtures, the fine aggregate used was a natural river sand with the specific gravity of 2.65, fineness modulus of 2.59, and water absorption of 1.5%. The physical properties and sieve analysis of aggregates are presented in Table 2 and Fig. 1, respectively.

The XRD analysis was used to characterize the mineralogical composition and some crystalline phases of the aggregates. The samples were prepared by drying in a vacuum drying machine at 50 °C for two days, and then grounded using ball mill until it passed a 125-μm sieve mesh. The XRD analysis was performed using multi-purpose X-ray diffractometer at a maximum operating power of 60 kV and 55 mA, with a scanning range of 3–60° 2θ. The XRD analysis patterns of ES, CRA, and RRA are presented in Fig. 2. The high-intensity peaks in the ES pattern indicate the presence of high amount of quartz, and the low-intensity peaks indicate muscovite and magnetite. Similarly, the RRA and CRA pattern also indicates the presence of quartz with higher intensities. The existence of portlandite, anhydrite, calcite and other aggregate minerals are also observed with small intensities. Rock aggregates usually consist several minerals with definite chemical compositions and specific crystalline structures [4]. For recycled aggregates, the contamination of the old cement paste by sand or original aggregates could be responsible for the higher-intensity of quartz and the lower-intensity of different mineral phases. Calcite is also observed with high-intensity for both CRA and RRA; however, the intensity of the calcite was higher for CRA than RRA.

TGA was used to measure the weight loss due to decomposition of different phases in the aggregates during heating. The samples were prepared in a similar manner to that for the XRD test and only the recycled aggregates (RRA and CRA) were tested by TGA. During measurement, the temperature was increased from 30 to 1000 °C at the rate of 10 °C per minute. The TG and DTG curves of RRA and CRA are presented in Fig. 3, where it is shown that the decomposition of calcite (CaCO₃) for both RRA and CRA occurred from 650 to 760 °C. Owing to the effect of carbonation, the mass loss from the decomposition of CaCO₃ in CRA (7.9%) was higher than that of CRA (5.1%). Based on the molar mass ratio, the content of calcite in CRA and RRA can be estimated as approximately 17.9% and 11.6% by weight, respectively [24]. The decomposition of Ca(OH)₂ was expected at approximately 400–500 °C [25]; however, this was not observed, with the exception of negligible waving. This might be attributed to the lower amount present as also shown in XRD analysis (Fig. 2).

The pore size distribution of the aggregates used in this experimental program was analyzed using mercury intrusion porosimetry (MIP) by applying a pressure from 0 MPa to 414 MPa (0 MPa to 60,000 psi). The surface tension, density, and contact angle of mercury were 0.485 N/m, 13.5335 g/ml, and 130°, respectively. The samples were prepared as a thin layer to avoid any test executed according to ASTM D4440 [26]. The pore size distribution and cumulative mercury intrusion result from the MIP analysis for all aggregates (RRA, CRA, and ES) are shown in Fig. 4(a) and (b), respectively. The results show that ES has a higher pore size, with an average median pore diameter of 979.5 nm, than those of CRA and RRA, which are 125.1 and 84 nm, respec-