Title (A novel method of crushing glass aggregates to reduce the alkali-silica reaction)

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Abstract (150 words)

This study proposed a novel method of crushing glass aggregates to reduce the alkali-silica reaction (ASR). Glass aggregates were crushed while immersed in different calcium bearing solutions. ASR measurement and compressive strength tests of mortars incorporating the glass aggregate were performed to investigate the feasibility of the crushing method. The results indicate that the crushing method was effective in both ASR mitigation and compressive strength improvement. Calcium nitrite solution was found to be more effective than calcium chloride solution for mitigating ASR damage. The ASR expansion after 14 days, measured in accordance with ASTM C1260, was 0.228% when the glass aggregates were conventionally crushed in air, which is potentially unsafe. This value decreased to 0.082% when the aggregates were crushed in calcium nitrite solution, a value which is considered innocuous. The compressive strength of the mortar was enhanced by approximately 12% compared to that of a mortar with conventionally crushed glass aggregate by incorporating glass aggregates crushed in calcium nitrite solution.

Keywords: Aggregates; alkali-silica reaction; glass aggregate; recycling; crushing

1. Introduction

Although more than 80% of waste glass is reused in the glass industry, it still causes a significant environmental problem because an enormous amount of glass is consumed in urban areas (Rahim et al. 2015). The construction industry has made efforts to recycle waste glass as a building material for decades. Numerous researchers have found that waste glass can be used as a cement and/or aggregate replacement. The effects of glass particle size are also reported (Cota et al. 2015; Du and Tan 2013; Xie et al. 2003; Rajabipour et al. 2010; Lee et al. 2011; Idir et al. 2010a; Yamada and Ishiyama 2005; Bažant et al. 2000; Shi et al. 2005; Kim et al. 2014, 2015; You et al. 2016; Zheng 2016; You et al. 2019; Kalakada et al. 2020). Incorporating glass particles as aggregates causes the alkali-silica reaction (ASR) to occur (Cota et al. 2015; Du and Tan 2013; Xie et al. 2003; Rajabipour et al. 2010; Lee et al. 2011; Idir et al. 2010a; Yamada and Ishiyama 2005), while fine glass powder is helps to mitigate ASR (Bažant et al. 2000; Shi et al. 2005; Kim et al. 2014, 2015; You et al. 2016; Zheng 2016; You et al. 2019; Kalakada et al. 2020) and increases resistance to chloride penetration (Kalakada et al. 2020; Chen et al. 2006; Shayan and Xu 2006; Tan and Du 2013; Guo et al. 2015; Kou and Poon 2009; Lee et al. 2018; Zidol et al. 2017; Wang et al. 2009; Jain et al. 2020). Most of the recent research is focused on the use of glass powder as a partial substitute for cement binder for various purposes (Jain et al. 2020; Kalakada et al. 2020; Duan et al. 2020; Mohammadyan-Yasouj and Ghaderi 2020; Shoaei et al. 2020; Rodier et al. 2020; Kadik et al. 2020; Adesina and Das 2020; Bostanci 2020). Although the use of glass in a powdered form is beneficial from the mechanical point of view, powdering the glass consumes a significant amount of energy, which increases the cost.

Recent studies found that a highly expansive ASR gel with a low Ca/Si ratio is formed in the residual cracks of a glass aggregate (Rajabipour et al. 2010). Clearly, such a gel would fracture the glass aggregate. Gholizadeh-Vayghan et al. (Gholizadeh-Vayghan and Rajabipour 2017b,a)

26 found that the expansibility of the ASR gel decreases with increasing Ca/Si ratio. ASR gels with

27 a Ca/Si ratio exceeding 0.5 did not cause any detrimental expansion.

28

29 Other experimental works found that the Ca/Si ratio of the ASR gel in the glass cracks is much

30 lower than that at the glass paste interface (Rajabipour et al. 2010; Shim 2019; Liaudat et al. 2019).

31 This is because of calcium ions originating from the paste neighboring the glass particles. The

32 chemical compositions of the ASR gel in the cracks and at the interface are consistent with the

33 availability of calcium ions in each region. A high Ca/Si ratio would help to decrease expansibility

34 of the gel between the crack surfaces, but any diffusive migration of calcium ions may not be

35 sufficient to increase the Ca/Si ratio.

36

37 This argument suggests artificially increasing the concentration of calcium ions in the residual

38 cracks of glass aggregates in advance of use as a partial aggregate substitute. After the crushing

39 procedure to produce glass aggregates, numerous residual cracks form on the surface of aggregate.

40 A high concentration of calcium ions may decrease the expansibility of the ASR gel in the residual

41 cracks. The higher the Ca/Si ratio is, the less expansive the ASR gel is. Furthermore, a gel with a

42 high Ca/Si ratio would seal residual cracks preventing the migration of harmful ions into residual

43 cracks.

44

45 In this paper, a novel method is proposed to reduce the ASR of glass aggregates. Instead of the

46 conventional procedure, i.e., crushing waste glass in air, the waste glass was crushed in a calcium

47 solution, and we refer to this process as the immersion-crushing (IC) process. Different types and

48 concentrations of calcium solutions were tested to investigate their effects on the mitigation of

49 the ASR. A control group was set up to compare the mitigation effect of the IC process on ASR.

50 In the control group, the waste glass was crushed in air before being immersed in the solution.

51   We refer to this process as the immersion-after-crushing (IAC) process. Finally, the effect of the

52   IC process on the compressive strength development was investigated.

53

54   There are several advantages of incorporating waste glass in construction materials by using the

55   immersion-crushing process proposed in this study. Normally, waste glass must be ground to a

56   fine powder to mitigate ASR, which consumes a significant amount of energy. The proposed

57   process makes it possible to recycle waste glass in concrete as aggregates, contributing not only

58   to the mitigation of the ASR, but also to improvement of the compressive strength, saving time

59   and resources.

60

61   **2. Experimental procedure**

62   *2.1 Materials*

63   Type-I Portland cement (PC) was used as the binder. The glass aggregates were produced by

64   crushing waste soda-lime glass (SG) bottles. The waste glass was obtained from a company which

65   collects domestic waste glass in Korea (Sungin 2019). Their chemical compositions are given in

66   Table 1.

67   Table 1: Chemical compositions of cement and glass aggregates.

Content (%)	CaO	Al_2O_3	SiO_2	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	LOI
PC	61.94	4.73	20.72	3.72	3.07	2.31	0.01	1.00	2.56
SG	9.49	2.82	67.13	0.01	1.83	0.01	14.01	0.8	0.30

68

69   It requires calcium compounds with high solubility to prepare high concentration calcium ionic

70   solutions. Calcium chloride, a widely used deicing salt, was used as a source of calcium. However,

71   it will bring chloride ions into concrete results in the corrosion of rebar. Alternatively, calcium

72 nitrite, an extensively used corrosion inhibitor (Al-Amoudi et al. 2003; Ann et al. 2006; Loulizi

73 et al. 2000; Song et al. 2019; Dong et al. 2018), was used as another source of calcium to

74 investigate the effect of calcium sources on the mitigation of ASR. 0.5 and 1.0 M calcium

75 solutions were prepared for each calcium compound, respectively, to study the effect of the

76 calcium concentration.

77

78 *2.2 Preparation of glass aggregates*

79 Two different processes were adopted to crush the waste glass into glass aggregates. The first was

80 crushing the solution-immersed glass, i.e., the immersion-crushing (IC) process. The second one

81 was crushing the glass in air and then immersing the aggregates in the solutions, i.e., the

82 immersion-after-crushing (IAC) process. A reference group was prepared by crushing waste glass

83 in air without immersion in or exposure to any solutions. A detailed description of the crushing

84 process is as follows.

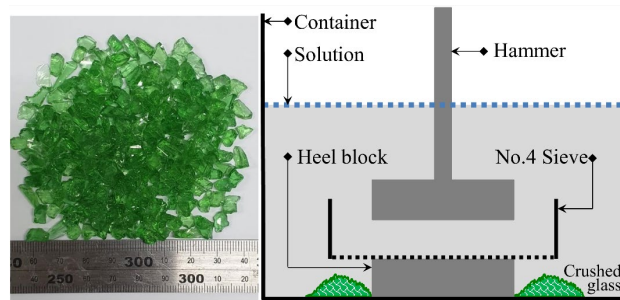85 *2.2.1 Immersion-crushing process*

86

87 Figure 1: Glass sample and crushing process of the waste glass.

88 After being washed and dried, the prepared glass bottles were crushed while immersed in each of

89 the respective calcium solutions. A schematic of the IC process is shown in Fig. 1. Only glass

90 particles that passed through a 4.75 mm square hole wire mesh sieve were collected.

91

4

92   The 0.5 and 1.0 M calcium nitrite and calcium chloride solution immersion-crushed glass particles

93   were denoted as IC-0.5CN, IC-1.0CN, IC-0.5CC, and IC-1.0CC, respectively.

94

95   Any solution remaining on the surface of the glass particles after the IC process may contribute

96   to the ASR mitigation. To check this contribution, the glass particles crushed in calcium nitrite

97   were washed using deionized water and denoted as IC-0.5CN-W and IC-1.0CN-W, respectively.

98   Glass particles crushed in deionized water were denoted as IC-H.

99

100   *2.2.2 Immersion-after-crushing process*

101   Water may quickly migrate into the residual cracks due to capillary action. Thus, the residual

102   cracks may also be filled with calcium solutions because of immersing the glass particles in the

103   solutions for a certain period after crushing in air, i.e., the IAC process. This leads to an expect

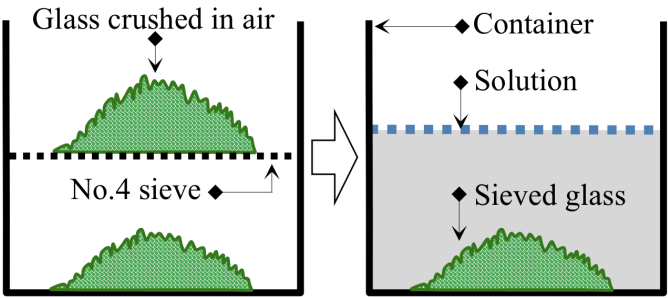104   action that the IAC process may result in the same degree of ASR mitigation as that of the IC

105   process.

106

108   Figure 2: Glass sample and crushing process of the waste glass.

109

110   A schematic of the immersion after crushing procedure is shown in Fig. 2. The waste glass bottles

111   were crushed in air, and only the particles passing through the 4.75 mm sieve were collected and

112 immersed in the prepared solutions for 30 minutes. The same solutions as mentioned in Section

113 2.2.1 were used, for comparative study.

114

115 The glass particles immersed in different concentrations of calcium nitrite and calcium chloride

116 solutions were denoted as IAC-0.5CN, IAC-1.0CN, IAC-0.5CC and IAC-1.0CC, respectively.

117 The particles immersed in the calcium solutions and then washed in deionized water were denoted

118 as IAC-0.5CN-W and IAC-1.0CN-W, respectively. The control group immersed in deionized

119 water was denoted as IAC-H.

120

121 *2.2.3 Grouping of test glass samples*

122 The glass particles were weighed after being collected from the solutions. The particles were then

123 moved to an oven preheated to 100.0 ∘ C and dried for 24 hours. The amount of solution on the

124 particle surface was measured by comparing the weights before and after drying.

125

126 The larger the glass particles are, the greater the ASR expansion (Idir et al. 2010a; Lee et al. 2011;

127 Du and Tan 2013; Rashad 2014). Glass particles in the range of 2.36 to 4.75 mm were chosen as

128 the aggregates of the test specimens.

129

130 The identification of the test samples follows that of the glass particles as presented in Table 2.

131

132 Table 2: Grouping and notation of the test samples.

Solutions	Immersion-crushing (IC)	Immersion-after-crushing (IAC)

Deionized water	IC-H	IAC-H
0.5 N $CaCl_2$	IC-0.5CC	IAC-0.5CC
1.0 N $CaCl_2$	IC-1.0CC	IAC-1.0CC
0.5 N $Ca(NO_2)_2$	IC-0.5CN	IAC-0.5CN
1.0 N $Ca(NO_2)_2$	IC-1.0CN	IAC-1.0CN
0.5 N $Ca(NO_2)_2$ *	IC-0.5CN-W	IAC-0.5CN-W
1.0 N $Ca(NO_2)_2$ *	IC-1.0CN-W	IAC-1.0CN-W
Crushed in the air	None	
All sand aggregates	Sand	

133   *Washed after immersion-crushing in the corresponding solution.

134

2.3 Specimen preparation and test methods

136   Mortar bars were prepared using the glass aggregates in accordance with ASTM C1260, as shown

137   in Fig. 3. Coarse aggregates were not included in this study. The mix proportion of water, cement

138   and sand was 0.47:1:2.25, where 20% by weight of the sand was replaced by glass aggregate. A

139   reference group was prepared only using sand that was denoted as "Sand". An accelerated ASR

140   test was performed in accordance with ASTM C1260. The average value of the results obtained

141   from three mortar samples was reported as the measure of the ASR expansion.

143   Figure 3: Mortar bars before demoulding for the ASR test.

144   The influence of the IC process on strength development of the mortars was evaluated by the

145   compressive strength test. Cubic mortar specimens were prepared in accordance with JGJ/T 70-

146   2009 using the same mixtures as those of the ASR test, as shown in Fig. 4. Nine sets of mortar

147   blocks were prepared. Each set comprised three samples. The compressive strength was measured

148   after 90 days curing at a temperature of 23 ± 2 °C and a relative humidity of more than 95% with

149   a universal testing machine. The average strength of the three mortar specimens having the same

150   mixture was recorded.

151   11 sets of mortar bars and mortar blocks were prepared. Each set comprised three samples.

152   mortar blocks ($70.7 \times 70.7 \times 70.7$ mm³) were prepared using the prepared glass aggregates.

153

154   Figure 4: Mortar blocks for the compressive strength test.

155

156

157   **3. Results and discussion**

158   *3.1 ASR expansion of immersion-crushing process glass aggregates*

159   The ASR expansion of the specimens incorporating glass aggregates obtained by the IC process

160   is shown in Fig. 5. The ASR expansion was slightly reduced when the glass was crushed in water

161   (IC-H). Kokura et al. (Kokura et al. 1989; Tomozawa et al. 1991) found that there were less

162   defects on the surface of glass particles crushed in water by electron spin resonance (ESR). When

163   the glass was crushed while immersed in calcium solution, the mitigation of ASR expansion was

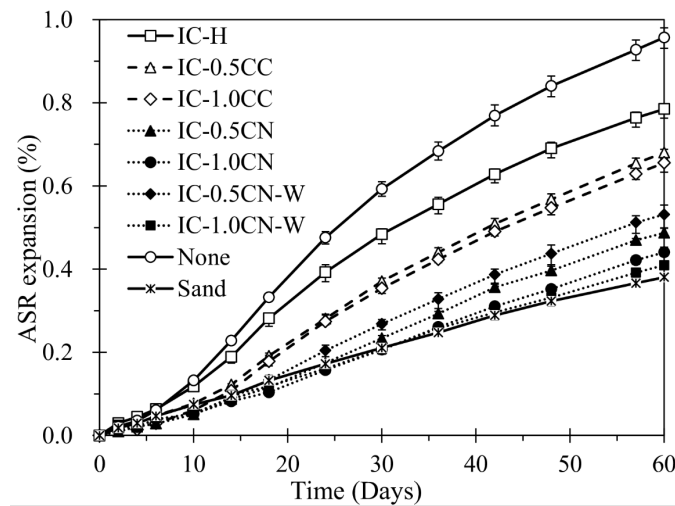164   remarkable for all the calcium solutions considered in this study.

165

166

167   Figure 5: ASR expansion of mortar bars incorporating the glass aggregates produced by

168   the IC process.

169

170   Calcium nitrite solution (IC-0.5CN and IC-1.0CN) mitigated ASR more effectively than calcium

171   chloride solution (IC-0.5CC and IC-1.0CC) as shown in Fig. 5. This improvement may be the

172   consequence of the accelerated hydration of cement by calcium nitrite (Li et al. 2000, 1999). The

173   cement particles in contact with the calcium nitrite in the cracks produce calcium silicate hydrate

174   (C-S-H) gel during the initial hydration stage. This C-S-H gel plugs the residual cracks and

175   decelerates the penetration of external alkali ions into the residual cracks, as schematically shown

176   in Fig. 6. As shown in Fig 6b, a protective layer is formed around the glass aggregate by the

177   hydrated particles, especially near the residual cracks, and consequently ASR expansion is

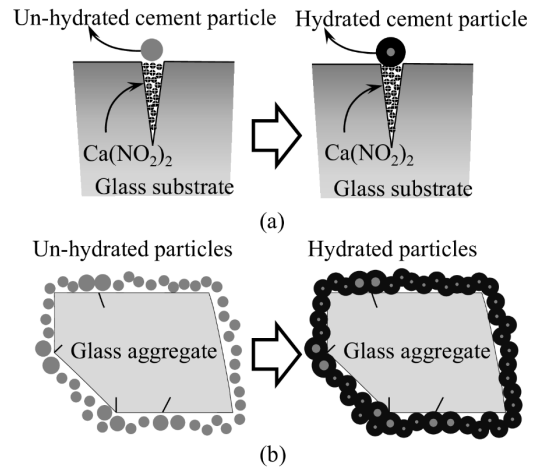178   reduced (Idir et al. 2010b; Maraghechi et al. 2017; Shafaatian et al. 2013).

179

Figure 6: Schematic description to explain the blocking effect of the C-S-H gel: (a) micro-scale, and (b) macro-scale.

A concentration of 0.5 M is sufficient to form this protective mechanism. The difference in the expansion was marginal when 1.0 M was tested as shown in Fig. 5. Although the optimum concentration was not determined, it can be seen from the sand line in Fig. 5, that the difference between 1.0 M calcium nitrite solution immersion-crushed glass and sand after 60 days is small.

Even after drying, there is residual calcium compound on the glass aggregate surface due to residual solution. This residual calcium compound is dissolved into the concrete matrix during mixing and may contribute to the mitigation of ASR. The content of the residual solution is shown in Fig. 7. The average solution content was approximately 5% by weight for both calcium chloride and calcium nitrite solutions. This is a considerably low value to mitigate ASR expansion (Kawamura and Ichise 1990; Li et al. 1999; Prezzi et al. 1998; Li et al. 2000). Furthermore, for IC-0.5CN-W and IC-1.0CN-W the effects of residual solution were eliminated by washing the aggregates in water after the IC process, as shown in Fig. 5, despite this however, the same trend of ASR mitigation was observed.
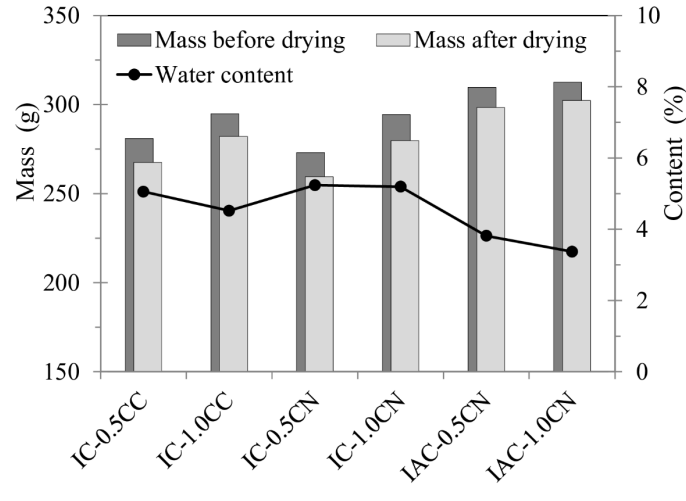
198

199   Figure 7: Content of the residual solution on the surface of the glass particles.

200

201   Fig. 8 shows the relative ASR expansion of the mortar bars normalized to the sample group

202   'None'. The reference samples incorporated only the conventionally crushed glass aggregates.

203   The figure shows that the ASR expansion was reduced by 20%, simply by crushing the glass in

204   water (IC-H). The ASR reductions when the aggregates were crushed in the calcium chloride (IC-

205   0.5CC, IC-1.0CC) and calcium nitrite solutions (IC-0.5CN, IC-1.0CN, IC-0.5CN-W, IC-1.0CN-

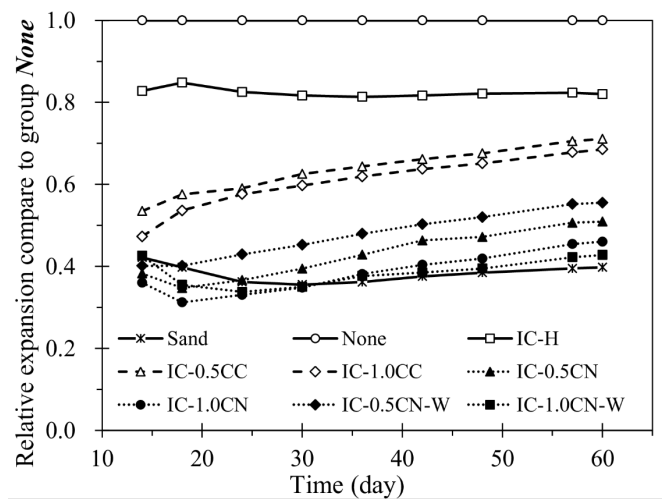206   W) were remarkably approximately 40% and 60%, respectively.

207

208   Figure 8: Relative ASR expansions of specimens compared to the control group, None.

209

210   The ASR expansion after 14 days is shown in Fig. 9. An expansion greater than 0.2% after 14

211   days is considered potentially unsafe and an expansion less than 0.1% innocuous, according to

212   ASTM C 1260. Therefore, the conventionally crushed glass ('None') presents an ASR risk.

213   Crushing the glass in calcium chloride solutions (IC-0.5CC, IC-1.0CC) effectively reduced the

214   expansion, but the expansion after 14 days still slightly exceeded 0.1%. The glass aggregates

215   crushed in calcium nitrite solutions (IC-0.5CN, IC-1.0CN, IC-0.5CN-W, IC-1.0CN-W) was

216   innocuous according to the ASTM C1260 criterion, despite some of them being washed after

217   crushing. Remarkably, the ASR expansion of three of the calcium nitrite samples was lower than

218   that of the specimens in which only sand was incorporated as aggregate ('Sand') after 14 days.
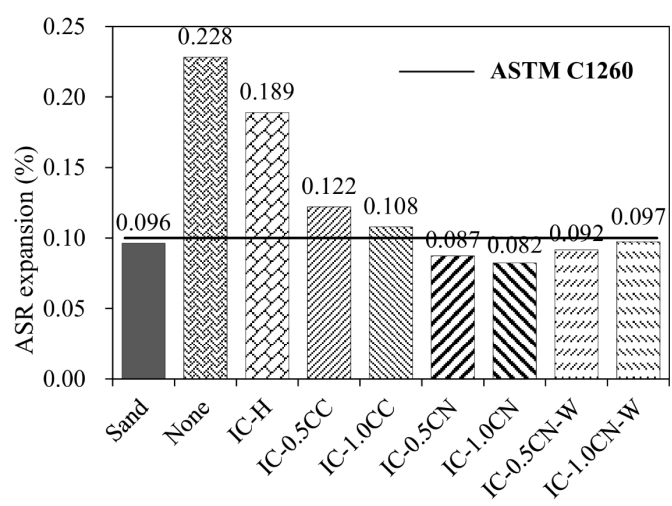

220   Figure 9: Effects of the crushing methods on the ASR expansion after 14 days.

3.2 ASR expansion of immersion-after-crushing prepared glass aggregates

223   Fig. 10 shows the ASR expansion of specimens incorporating the glass aggregates prepared by

224   the IAC process. It is interesting to note that the immersion of glass particles in the solutions after

225   they were crushed in the air did not affect the ASR expansion. The expansion after 14 days is

226   about 0.2%, regardless of the solution in which particles were immersed. The difference in ASR

227   expansion according to different conditions was insignificant, and even after 60 days, only ranged

228   from 0.88% to 0.95%.

229

230   There are two possible explanations for the marginal effect of the IAC process on ASR expansion.

231   The IAC process did not affect the physical characteristics of the glass particles, such as reducing

232   the surface defects resulting from the IC process. Most of the residual cracks in the glass particles

233   had closed channels at one end with widths of approximately 2-5 μm (Maraghechi et al. 2012).

234   The capillary action of closed-end nano-channels is limited because of the gas pressure inside the

235   capillary (Radiom et al. 2010; Lim et al. 2014, 2019). Thus, it is difficult for calcium ions to

236   ingress into cracks solely by immersion for 30 minutes after the glass aggregates have been

237   crushed in air.
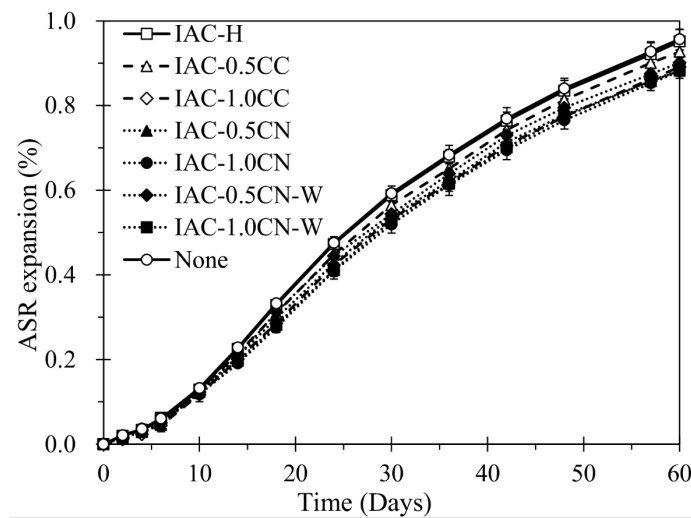
238

239   Figure 10: ASR expansion of mortar bars incorporating the glass aggregates produced by

240   the IAC process.

241

242   *3.3 Appearance of the test samples after 60 days*

243

244   The appearance of the mortar bars after 60 days of accelerated ASR testing is shown in Fig. 11.

245   The results agree well with the ASR expansion results shown in Figs. 5 and 10. A small number

246   of visible cracks were observed in the specimens incorporating the aggregates prepared using the

247   IC process. Almost no visible cracks were observed on the surface of the specimens incorporating

248   the aggregates crushed in the calcium nitrite solutions (IC-0.5CN and IC-1.0CN). This confirms

249   that crushing the glass aggregates in calcium nitrite solution can effectively mitigate ASR

250   expansion. A number of cracks were

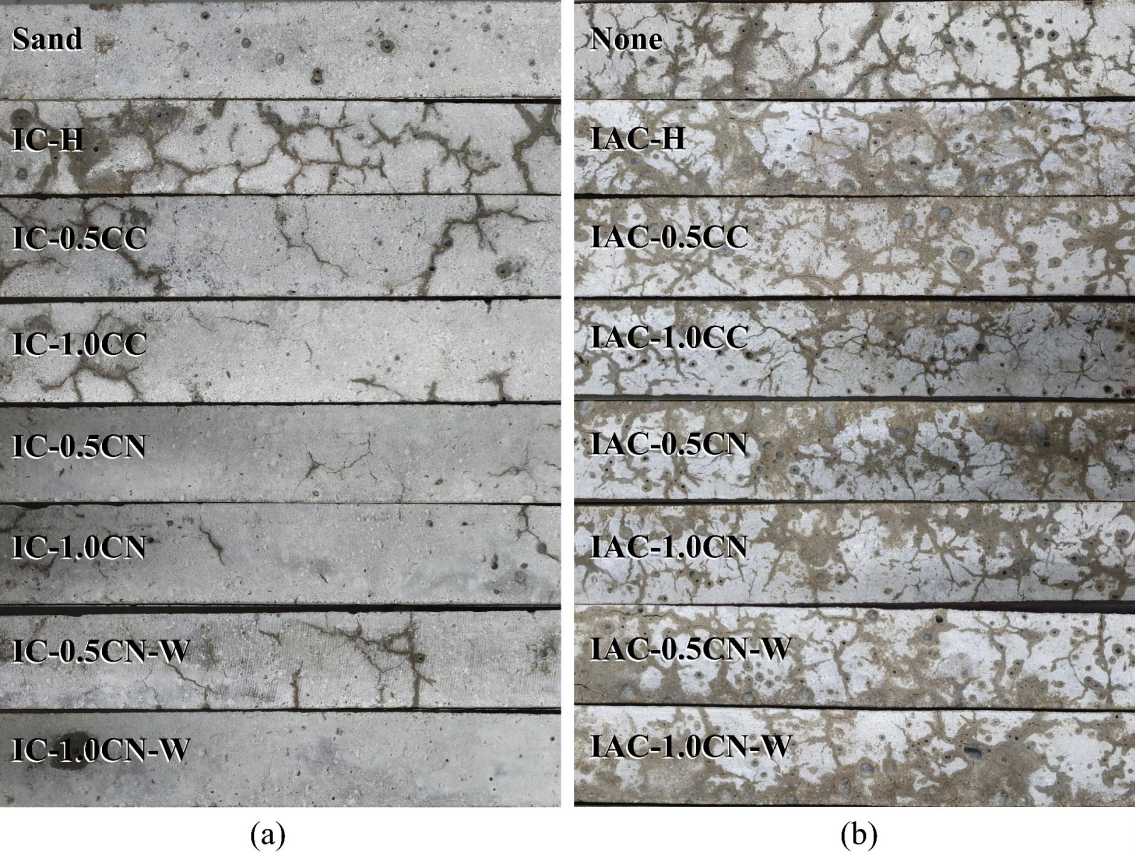251   observed in the specimens which contained glass aggregates produced by the IAC process.

(a)   (b)

253   Figure 11: Photos of mortar bars subjected the accelerated ASR test for 60 days: (a) IC,

254   and (b) IAC.

255

256

257   *3.4 Effect of the immersion-crushing process on the compressive strength*

258

259   The compressive strength of specimens incorporating glass aggregates crushed in different

260   solutions is shown in Fig. 12. The compressive strength was reduced by about 6.0% by replacing

261   20% of the sand with the conventionally crushed glass aggregates ('None'). Crushing the glass in

262   water (IC-H) or calcium chloride solutions (IC-0.5CC, IC-1.0CC) has no measurable effect on

263   the compressive strength. However, crushing the glass in calcium nitrite solution (IC-0.5CN, IC-

264   1.0CN) increased the compressive strength by about 12%. This compressive strength even

265   exceeds that of the 'Sand' group. The compressive strength was slightly decreased by washing

266   the glass aggregates after they were crushed in the calcium nitrite solution (IC-0.5CN-W, IC-

267   1.0CN-W). While, similar to the ASR results, the concentration of the calcium solution had no
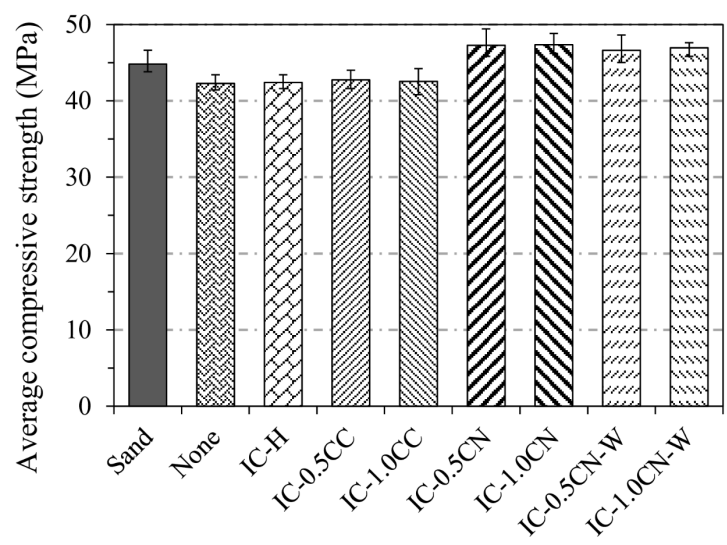
268   noticeable effect on the compressive strength.

269

270

271   Figure 12: Effect of the immersion-crushing process on the compressive strength after 90

272   days of curing.

273

274   **5. Conclusions**

275   Glass aggregates were crushed while immersed in different calcium solutions. The effects of this

276   novel crushing process on ASR and compressive strength were investigated experimentally. The

277   conclusions of this research are summarized as follows:

278

279   (1). ASR of glass aggregates can be dramatically mitigated by crushing them while immersed in

280   calcium solutions. However, the effect is marginal if the crushed glass aggregates are immersed

281   in calcium solutions after being crushed in air.

282

283   (2). Calcium nitrite solution is more effective than calcium chloride solution for ASR mitigation.

284   ASR expansion mitigation was approximately 40% and 60% for calcium chloride and calcium

285   nitrite solutions, respectively.

286

287   (3). The mortar bars incorporating glass aggregates crushed in calcium nitrite solution exhibited

288   innocuous ASR behaviour according to ASTM C1260 criterion, i.e., ASR expansion after 14 days

289   is less than 0.1%.

290

291   (4). The compressive strength of specimens incorporating glass aggregates crushed in calcium

292   nitrite solution was increased. Its value after 90 days of curing was approximately 12% higher

293   than that of specimens incorporating conventionally crushed glass aggregates, and approximately

294   5.3% higher than the specimens with exclusively sand aggregate.

295

296

297   **Acknowledgements**

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