Expansion due to alkali-silica reaction of ferronickel slag fine aggregate in OPC and blended cement mortars

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Abstract

About 14 tonnes of granulated ferronickel slag (FNS) is produced as by-product in the production process of 1 tonne of ferronickel. The physical properties of FNS are suitable for its use as fine aggregates in concrete. This paper presents a study on the expansion due to alkali-silica reaction (ASR) of mortar bars containing different percentages of FNS aggregates originated from garnierite ore. Accelerated mortar bar test (AMBT) was performed to measure the expansion of the samples. FNS was found to be reactive in Portland cement mortar according to the AMBT results. Class F fly ash and ground granulated blast furnace slag (GGBFS) were used as the ASR mitigating measures of the FNS aggregate. Experimental results show that the use of GGBFS was unable to reduce expansion to the required level. However, the use of 30% class F fly ash as a supplementary cementitious material was found to be effective in reducing the 21-day ASR expansion below 0.3% required by the Australian Standard. Observations by scanning electron microscopy confirmed the effectiveness of fly ash in mitigation of the ASR products of FNS aggregates.

1. Introduction

Laterite, garnierite and pentlandite ores are the primary sources of nickel. The ore is smelted in an electric furnace or rotary kiln in order to produce ferronickel. The granular slag comes out as a by-product of the production process of ferronickel. The slag is classified into three types, depending on its cooling method such as slow-cooling granulated, wind granulated and water granulated [1]. The ore contains low grade nickel and it undergoes through three major stages such as pre-reduction, reductive smelting and ferronickel enrichment in the process of producing ferronickel. A large quantity of slag is produced in the process of ferronickel production. Safe disposal of the slag is considered as a major issue for the ferronickel plants worldwide [2]. Almost 14 tonnes of slag is generated per tonne of ferronickel production [3]. On the other hand, in order to prevent the rapid depletion of natural fine aggregates, the use of industrial by-products and recycled aggregates in concrete has been studied extensively for the past few decades. The Japan Industrial Standard (JIS) 5011-2 [4] has produced recommendations for using FNS as aggregates in concrete. Sato et al. [5] and Sakoi et al. [6] showed that replacing natural fine aggregate with air granulated FNS produced in Japan exhibited higher compressive strength and modulus of elasticity of concrete. Shoya et al. [7] and Sugita et al. [8] reported that 50% replacement of aggregates by the FNS exhibited better durability properties as compared to the control specimens. The study evaluated the permeability, carbonation and drying shrinkage properties of concrete. Though slags produced in different smelters may appear similar physically, their chemical compositions can vary significantly depending on the source of the ore and the manufacturing process. It is also essential to investigate the potential of alkali-silica reaction (ASR) of an aggregate. This is

because certain aggregates can cause expansion due to ASR depending on their chemical compositions. Deleterious ASR may result in large expansion causing concrete structures to crack.

Stanton [9] identified that concrete structure can suffer from the deleterious expansion due to ASR. Cole et al. [10] reported surface cracking and expansion of old dams in Australia suffering from ASR. The mechanism of ASR is complicated and there are differences in opinions among the researchers. According to Lee [11], the reactive silica in aggregate is depolymerised in a highly alkaline condition. Hydrolysis of the silica takes place in the presence of sodium and potassium hydroxides and an alkali silicate gel is formed. This gel absorbs a large quantity of water from the surroundings resulting in an expansion of its volume. This expansion of the alkali-silica gel causes micro-cracking inside the aggregate as well as at the interface between aggregate and cement paste [12]. Usually, aggregates with amorphous structure, lattice defects and large surface area are more susceptible to ASR expansion [13]. The crystalline structure of silica is stable and the amorphous silica is considered to be reactive [14]. The presence of calcium is also regarded as an important factor in ASR expansion. The calcium content of the binder has a significant influence on the expansion due to ASR. The ASR expansion increases with the rise of calcium content in the binder [15]. Chatterji [16] showed that the expansion could be reduced by leaching out of the $Ca(OH)_2$ from concrete. It was also observed that the reactive silica did not cause any harmful expansion if there was no calcium available in the binder [17]. The presence of a higher percentage of calcite was also found to cause greater expansion of concrete [18]. Since ASR is a slow process, accelerated tests of mortar or concrete specimens are usually conducted to assess the alkali reactivity of an aggregate. Such accelerated tests are recommended in testing Standards and Codes. A correlation between the accelerated ASR test results and the actual ASR in concrete structures was shown by Carse and Dux [19].

75   Use of supplementary cementitious materials (SCM) such as fly ash, blast-furnace slag

76   and silica fume is usually considered as a mitigating measure against the ASR expansion of

77   concrete. However, the effectiveness of an SCM in reducing the ASR depends of the type and

78   dosage of the SCM, and the type of the reactive aggregate. The effectiveness of an SCM

79   depends on its alkali content, with higher alkali content exhibiting higher expansion [20].

80   Shehata and Thomas [21], and Shafaatian et al. [22] showed that expansion of mortar

81   specimens containing reactive aggregates was reduced by the use of fly ash as a partial

82   replacement of cement. Fly ash can significantly reduce the alkalinity of pore solution resulting

83   in a reduction of the ASR [23]. The addition of reactive aggregates in powder form with the

84   binder can also reduce the ASR as the powder acts as a pozzolana and reduces the alkalinity of

85   the pore solution [24]. Although ASR is being studied for the past few decades, the exact

86   chemical reactions that cause the expansion and cracking of concrete remains in shadow.

87   The ASR expansion of FNS fine aggregate produced in the smelters of New Caledonia

88   has been investigated in this study. This particular FNS is extracted by pyro-metallurgy from

89   garnierite ore and cooled down by sea water. It has different chemical compositions than the

90   FNS aggregates used in the studies available in literature. Our other study [25] investigates the

91   utilisation of the ground FNS as a partial replacement of cement. Petrographic analysis of the

92   FNS showed the existence of glass containing amorphous and cryptocrystalline silica that

93   might cause ASR when used as aggregates in concrete. Therefore, a study on the potential ASR

94   expansion of the aggregate is essential for its development as an alternative fine aggregate for

95   concrete. Accelerated ASR tests were conducted on mortar specimens containing different

96   percentages of FNS as a replacement of natural sand. Fly ash and GGBFS were used as partial

97   replacements of cement and their effectiveness on the ASR expansion were studied.

98   Microstructural analysis using scanning electron micrographs (SEM) and energy-dispersive X-

99   ray spectroscopy (EDS) was performed to obtain an insight into the ASR products and the

100   resulting expansions observed for the mortar mixtures containing different percentages of the

101   FNS and SCMs.

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103   **2. Experimental work**

104   **2.1 Materials**

105   The materials used in this study were ordinary Portland cement (OPC), FNS, class F

106   fly ash and GGBFS. The chemical compositions of these materials are given in Table 1. It can

107   be seen that the main components of the FNS were SiO_2, MgO and Fe_2O_3. The CaO content of

108   the fly ash was very low (0.6%) as compared to that of GGBFS (41.2%). The SiO_2 content of

109   the fly ash (76.4%) was much higher than that of GGBFS (32.5%). Furthermore, Na_2O content

110   was less in fly ash (0.19%) than in OPC (0.29%) and GGBFS (0.27%).

111

112   Table 1 Chemical compositions and loss on ignition (LOI) of OPC, FNS, fly ash and GGBFS

113   (mass %)

Material	OPC	FNS	Fly ash	GGBFS
SiO_2	20.29	52.52	76.34	32.45
Al_2O_3	5.48	2.33	14.72	13.56
Fe_2O_3	2.85	10.80	3.69	0.82
MgO	1.24	33.16	0.54	5.10
SO_3	2.49	0.06	0.11	3.20
CaO	63.11	0.27	0.60	41.22
Na_2O	0.29	0.12	0.19	0.27
K_2O	0.45	0.04	0.96	0.35
Cr_2O_3	0.02	-	-	-
P_2O_5	0.17	0.01	0.10	0.03
SrO	0.05	-	-	-
TiO_2	0.27	0.04	0.61	0.49
Mn_2O_3	0.08	0.49	0.07	0.25
ZnO	0.04	-	-	-
LOI	3.39	0.01	0.53	1.11

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Figure 1 shows a comparison between the physical appearances of the FNS and natural sand. It can be seen that FNS aggregates comprised of angular particles of varying sizes, whereas, natural sand is spherical and of much more uniform size. Physical properties of the fine aggregates are given in Table 2. The fineness modulus of the FNS was higher than that of the natural sand.

Figure 1. Granulated FNS (left) and natural sand (right).

Table 2 Physical properties of natural sand and FNS

Property	Sand	FNS
SSD density (kg/m3)	2.16	2.78
Apparent particle density (kg/m3)	2.32	2.85
Fineness modulus	1.95	4.07

The grain size distributions of the natural sand, FNS and their combinations are shown in Figure 2. The upper and lower limits of the grading requirements recommended by the Australian Standard AS 2758.1 [26] are also plotted in this figure. It can be seen that the particle size distributions for all the combinations are within the recommended limits of the AS 2758.1, except for 100% FNS, for which the distribution is slightly beyond the lower limit between 300 µm and 1.8 mm sizes. It is also evidenced that the grading curve becomes more smooth and

132 continuous when the FNS and natural sand are mixed together. The most well-graded particle

133 size distribution was obtained for the combination of 50% FNS with 50% natural sand.
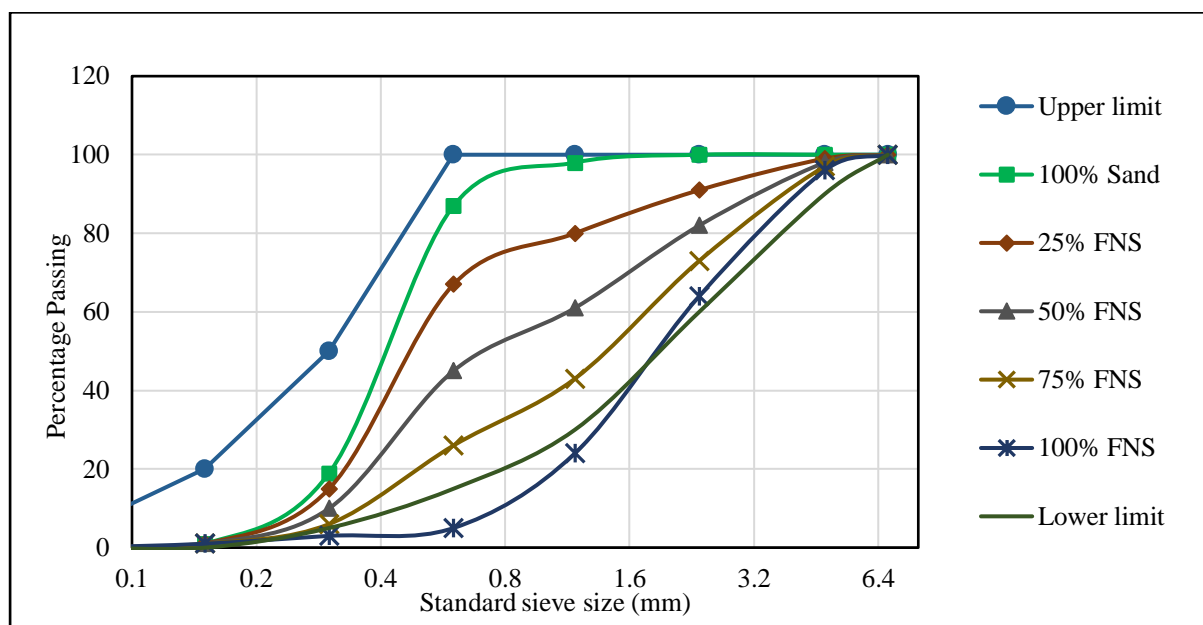
136 Figure 2. Particle size distributions of natural sand, FNS, their combinations, and the upper

137 and lower limits of AS 2758.1 [26].

2.2 Specimens and test methods

140   Petrography and expansion tests are two most common tests used for identification of

141 the alkali reactivity of aggregates. However, petrography test has the limitation of its efficiency

142 in identifying the fine-grained reactive silica [27]. Accelerated mortar bar test (AMBT) was

143 performed in this study, as it has been well accepted as a quick and efficient test to determine

144 the alkali reactivity of fine aggregates. Since the aggregates slow reactivity might not come

145 into reaction within a short period of 21 days, the test can be continued for a longer period of

146 time [28]. Therefore, the test was extended up to 64 days in this study. The tests were conducted

147 in accordance with the AS 1141.60.1 Standard [29]. The mortar bars were cast from mixtures

148 with a water to binder ratio of 0.47 and binder to fine aggregate ratio of 1 : 2.25, as

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recommended in the Standard. The test specimens were made from mortar mixtures with FNS

replacing natural sand by 25%, 50%, 75% and 100%. Fly ash and GGFS were used as

replacements of cement by 30% in order to study their effectiveness on mitigation of the ASR

of FNS. A 30% cement replacement was selected as this percentage is considered to give a

substantial reduction in the CO_2 emission with a reasonable reduction in the strength of

concrete [30]. The mixture proportions of the mortars are given in Table 3. The binder of the

mixtures of Series A was 100% OPC and those of Series B and C had 30% fly ash and 30%

GGBFS respectively.

Table 3 Mixture proportions of mortars

Mix ID	Binder (Kg/m^3)			Fine aggregate (Kg/m^3)		W/C
	OPC	FA	GGBFS	Sand	FNS	
A1	602	-	-	1355	-	
A2	602	-	-	1015	338	
A3	602	-	-	678	678	
A4	602	-	-	338	1015	
A5	602	-	-	-	1355	
B1	421	181	-	1355	-	
B2	421	181	-	1015	338	0.47
B3	421	181	-	678	678	
B4	421	181	-	338	1015	
B5	421	181	-	-	1355	
C1	421	-	181	1355	-	
C2	421	-	181	1015	338	
C3	421	-	181	678	678	
C4	421	-	181	338	1015	
C5	421	-	181	-	1355	

Mortar bar specimens of $25 \times 25 \times 285$ mm were cast with gage studs inserted at the

ends to measure the length change of the specimen. The specimens were cured at room

temperature for 24 hours and then they were submerged in a water bath at 80 $^\circ$C for one day,

after which the initial measurement of the length was taken. Then, the samples were submerged

in a bath of 1 mol/L NaOH solution. The first measurement of the change in length was

164 conducted after three days of immersion using a digital length comparator with an accuracy of

165 0.001 mm. The measurements were then continued at three days of intervals. A mortar bar

166 specimen and measurement of its length change are shown in Figure 3. Measurements were

167 taken within 10 seconds of removing samples from the solution.

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169

170 Figure 3. Length measurement of a mortar bar specimen.

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172 **3. Results and Discussion**

173 *3.1 Mortar bar expansion*

174 The changes in the expansion of the mortar bars with increasing immersion period for

175 the mixtures of 100% OPC (Series A) are shown in Figure 4. It can be seen that the expansion

176 is relatively small in the specimen with 100% natural sand. However, expansion increased

177 significantly with the inclusion of FNS in the mixture. The high expansion of the specimens is

178 attributed to the presence of amorphous silica in the FNS. Petrographic examination showed

179 that the FNS contained 44% amorphous and cryptocrystalline silica structures. Since the

180 amorphous silica is reactive with the free alkali present in the binder matrix, the FNS showed

181 high expansion due to the alkali-silica reaction. There was substantial increase of expansion in

182 the specimens with 25% and 50% FNS. The expansion increased at relatively smaller rates

with further increases in the percentage of FNS. The expansion of the specimen with 100%

natural sand increased gradually over the period of immersion. On the other hand, the

expansion of the specimens with 25% to 100% FNS increased at a high rate until 14 days and

then continued to increase at slower rates until 64 days. The expansions after 64 days of

immersion were 0.21%, 0.55%, 0.93%, 1.06% and 1.09% for the FNS contents of 0%, 25%,

50%, 75% and 100% respectively. Thus, it can be seen that there are only small increases in

the 64-day expansions by the increase in FNS above 50% up to 100%. This trend is similar for

the earlier periods of immersion.
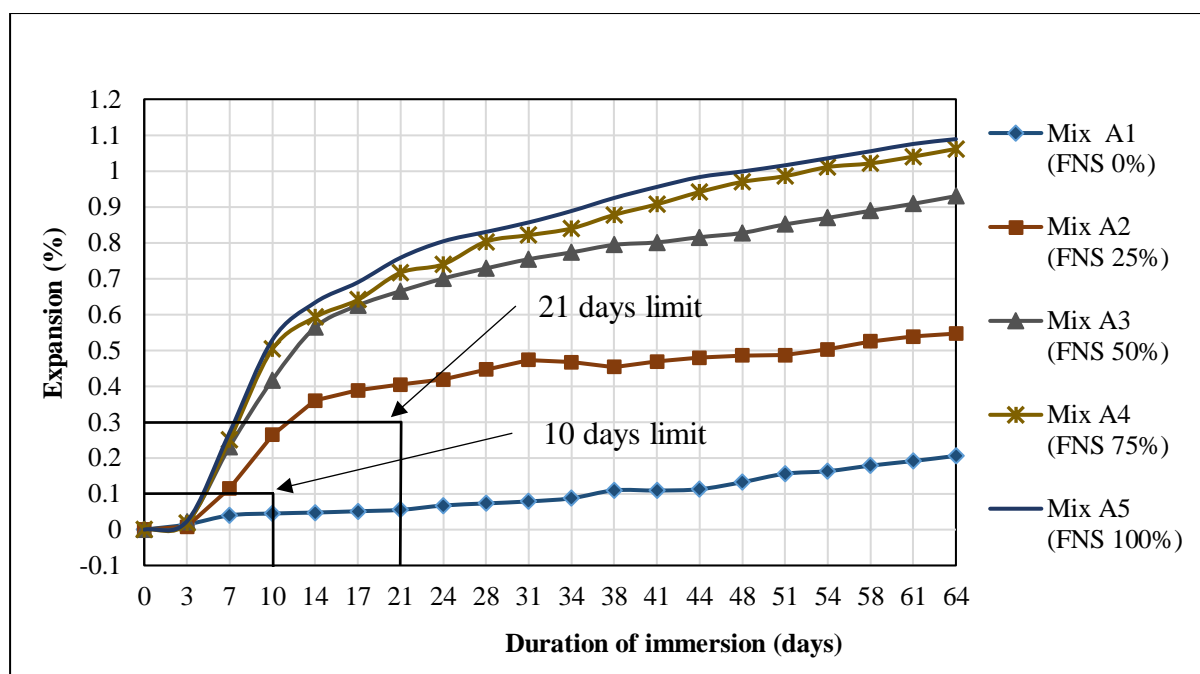
Figure 4. ASR expansions of mixtures of 100% OPC (Series A).

The reactivity classification according to the Australian Standard AS 1141.60.1 [29] is

reproduced in Table 4 in order to compare with the AMBT results for different mixtures

obtained in this study. According to this table, an aggregate should be classified as reactive if

the expansion exceeds 0.30% within 21 days of immersion. Appropriate measures are required

to reduce the expansion if reactive aggregates are to be used in concrete. The expansions of all

the fifteen mixtures of this study after 21 days of immersion are collected in Table 5. Each

mixture classified as reactive (R) or non-reactive (NR) by using the limits given in Table 4 are

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shown in Table 5. As shown in Fig. 4, the 14-day expansion of the specimen containing no

FNS (Mix A1) is 0.05%, which is much smaller than the 0.3% limit set by the Standard.

Therefore, the natural sand is classified as non-reactive. On the other hand, the 14-day

expansions of the specimens containing 25% to 100% FNS are higher than the 0.3% limit.

Therefore, the FNS is classified as a reactive aggregate in accordance with AS 1141.60.1 [29].

According to the Australian Standard AS 2758.1 [26], some ASR mitigating measure is

required for this FNS if it is to be used as an aggregate for concrete. Therefore, 30% fly ash

and 30% GGBFS were used as SCMs, and their effects on the expansions of the mixtures

containing 25% to 100% FNS were studied.

Table 4 Limits of AMBT expansion for reactivity classification as per AS1141.60.1 [29]

Mean mortar bar expansion (E), %		Aggregate reactivity classification
Duration in 1M NaOH at 80 °C		
10 days	21 days	
—	E < 0.10	Non-reactive
E < 0.10	$0.10 \leq E < 0.30$	Slowly reactive
$E \geq 0.10$	—	Reactive
—	$0.30 \leq E$	Reactive

Table 5 Expansion of mortar bars after 21 days in 1M NaOH solution at 80 ^{o}C

Binder Composition	Percentage of FNS as fine aggregate (%)				
	0	25	50	75	100
100% OPC (Series A)	0.06	0.40	0.66	0.72	0.76
reactive classification	NR	R	R	R	R
30% Fly ash + 70% OPC (Series B)	0.02	0.03	0.05	0.05	0.06
reactive classification	NR	NR	NR	NR	NR
30% GGBFS + 70% OPC (Series C)	0.04	0.34	0.63	0.69	0.72
reactive classification	NR	R	R	R	R

NB: NR= non reactive; R= reactive

The expansion results of the mixtures with 30% fly ash replacing cement (Series B) are

presented in Figure 5. Slight initial shrinkage was observed in the specimens of 0 to 50% FNS.

215 This is attributed to the initial autogenous shrinkage and low expansion by the use of fly ash

216 replacing 30% cement. It can be seen that the expansions of the mixtures containing 30% fly

217 ash are much lower than the values for the mixtures of 100% OPC. For example, the maximum

218 64-day expansion in this series is 0.09% for the mixture with 100% FNS. This value is much

219 smaller than the expansion (1.09%) for the mixture with 100% FNS and 100% OPC. This

220 shows that 30% fly ash as cement replacement has significantly reduced the expansion due to

221 ASR in the mortar specimens containing 25% to 100% FNS. While the expansions of all the

222 mixtures are seen to increase with increasing duration of immersion, the rates of increase are

223 seen to be negligibly small after immersion for 64 days. As shown in Table 3, the expansions

224 varied between 0.02% and 0.06% for 0% to 100% FNS. These values are much smaller than

225 the 0.3% limit of the Australian Standard [29]. Therefore, the replacement of cement by 30%

226 class F fly ash can be considered as an effective ASR mitigating measure for concrete using

227 FNS up to 100% replacement of natural sand.
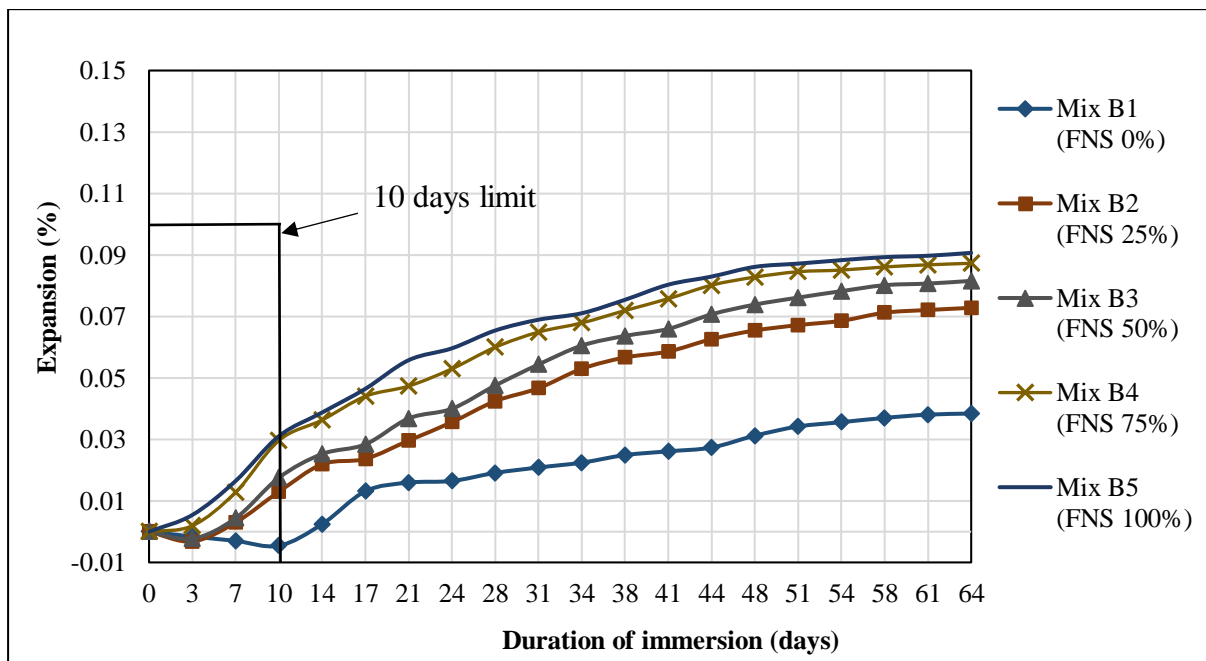
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229 Figure 5. ASR expansions of the mixtures of 70% OPC and 30% fly ash (Series B)

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The expansions of the specimens containing 30% GGBFS as cement replacement (Series C) are shown in Figure 6. It can be observed that the expansions of the specimens of this series are higher than those using fly ash as cement replacement. The higher expansions of these mixtures are attributed to the higher CaO content of GGBFS (41%) as compared to that of fly ash (0.6%). The higher CaO increased the total alkali content of the binder as compared to the mixtures with fly ash. Thus, the expansions due to ASR are higher in the mixtures with GGBFS than those of the mixtures with fly ash. The specimens with FNS continued to expand for immersions beyond 21 days, and the expansion became almost stable after 64 days of immersion. The 64-day expansion varied from 0.53% to 0.98% for the FNS content varying between 25% and 100%. Similar to the mixtures of other two series, there has been very small increase in the expansion for the increase in the percentage of FNS beyond 50%.
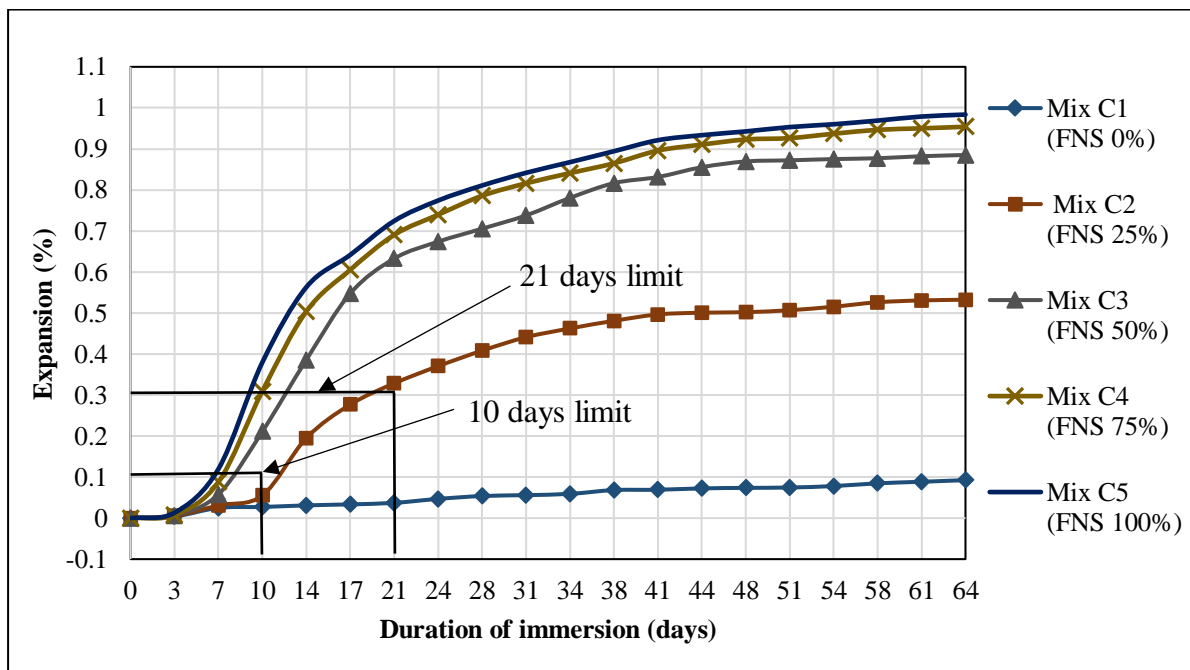
Figure 6. ASR expansions of mixtures with 70% OPC and 30% GGBFS (Series C)

The expansions of the specimens after 21 and 64 days of immersion are plotted against the percentage of FNS as replacement of natural sand in Figure 7. The 0.3% limit on the

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expansion after 21 days of immersion is also shown in this figure. It can be seen from the figure

that the expansions of the specimen containing 30% fly ash are below this limit for the mixtures

up to 100% FNS. Expansion of the specimen with 25% FNS and 30% GGBFS just exceeded

the limit of the Australian Standard [29] marginally and further increased with the increase of

the percentage of FNS. However, the increase in expansion for the specimens of 75% and 100%

FNS were very small as compared to that with 50% FNS. This trend is similar to the mixtures

with 100% OPC, 30% fly ash and 30% GGBFS. The increase in the expansion for the FNS

content beyond 50% is very small because there is less free alkali available for reaction with

the additional FNS aggregates. Therefore, it can be concluded that 30% fly ash effectively

reduced the expansion below the limit of the Australian Standard when FNS was used as a

replacement of natural sand by up to 100%. However, the reduction of expansion by 30%

GGBFS as cement replacement for FNS content of 25% and above was not enough to meet the

limit of the Australian Standard.
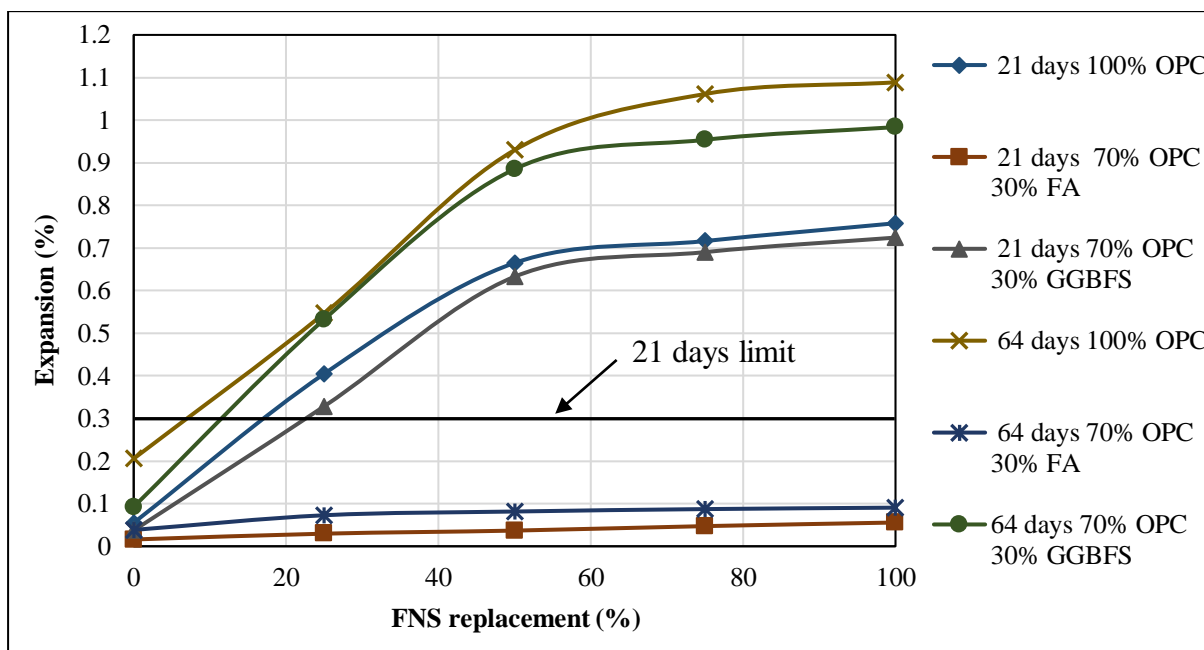
Figure 7. Variation of expansion with the percentage of FNS

3.2 Study of the microstructure by SEM and EDS

266   The specimens containing 100% FNS aggregates from the 3 series of mixtures were

267   selected to study the microstructure of the reaction product by using SEM photographs and

268   corresponding EDS data. The microstructures of the specimens containing 100% natural sand

269   were also investigated to compare with those containing FNS aggregate. Figure 8 presents the

270   SEM images and the corresponding EDS data for the mixtures A1, A5, B1, B5, C1 and C5.

271   Generally, foil-like crystals, rosette-like accumulation and hexagonal acicular crystals are the

272   most frequent types of ASR products found in SEM imaging [26]. Some of these distinct

273   features can be observed in the SEM images of the specimens containing FNS aggregates,

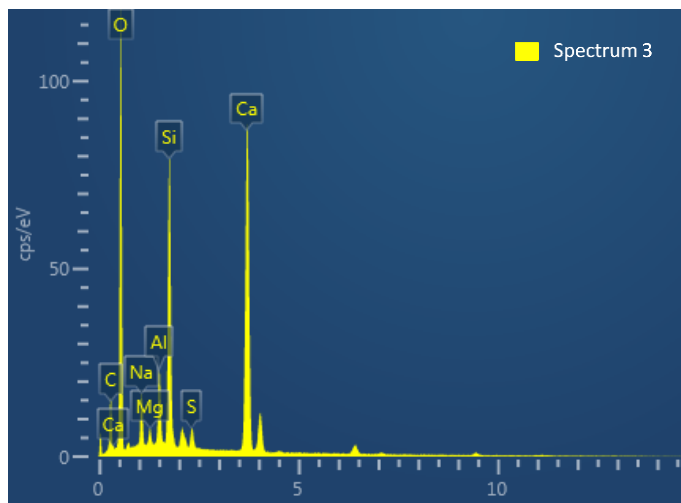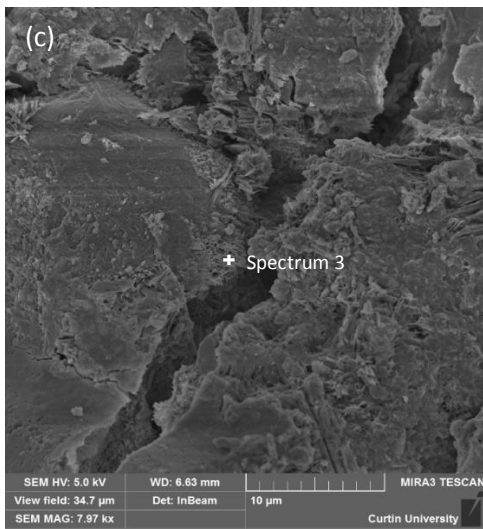274   especially those using 100% OPC, and 30% GGBFS and 70% OPC.
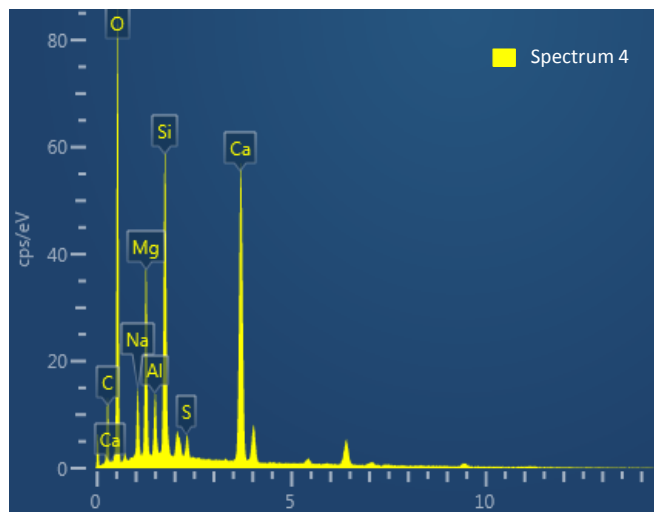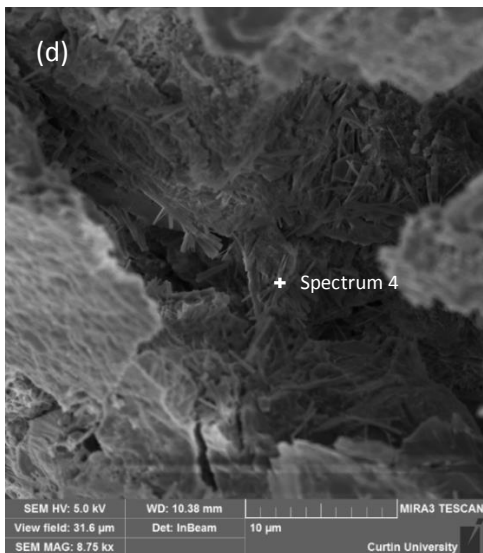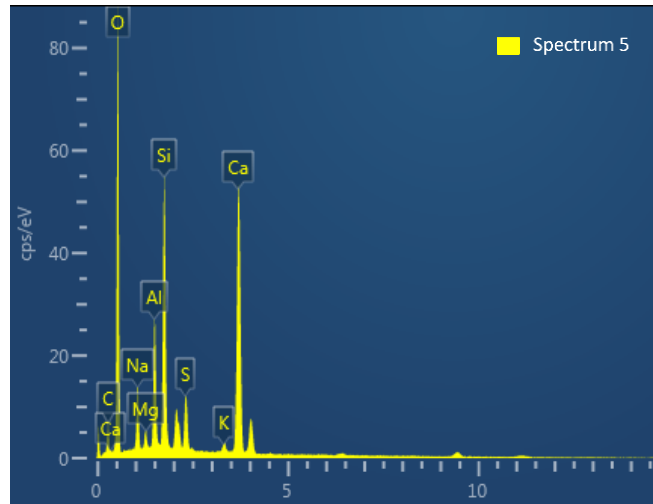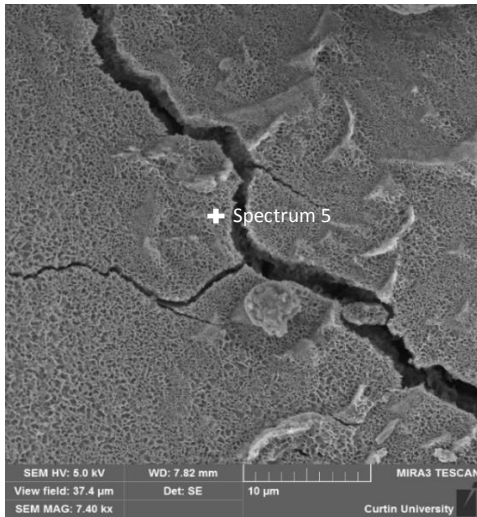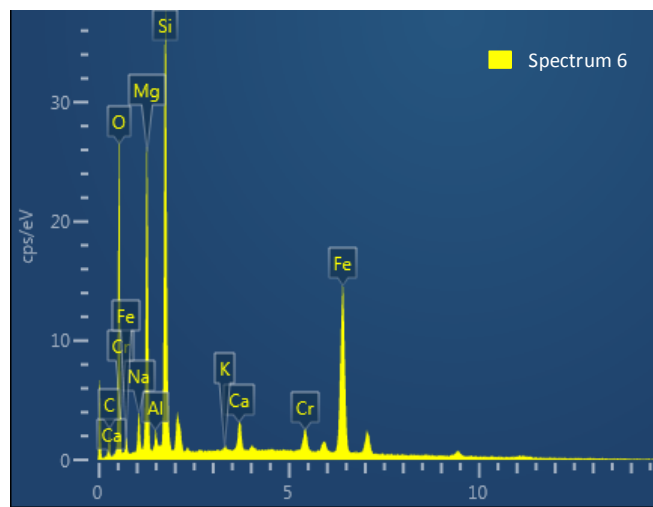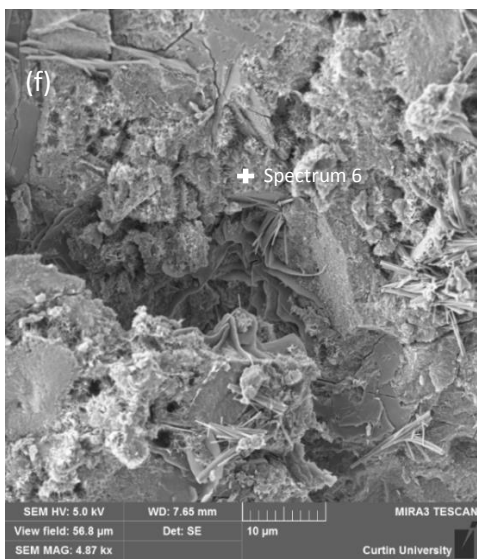
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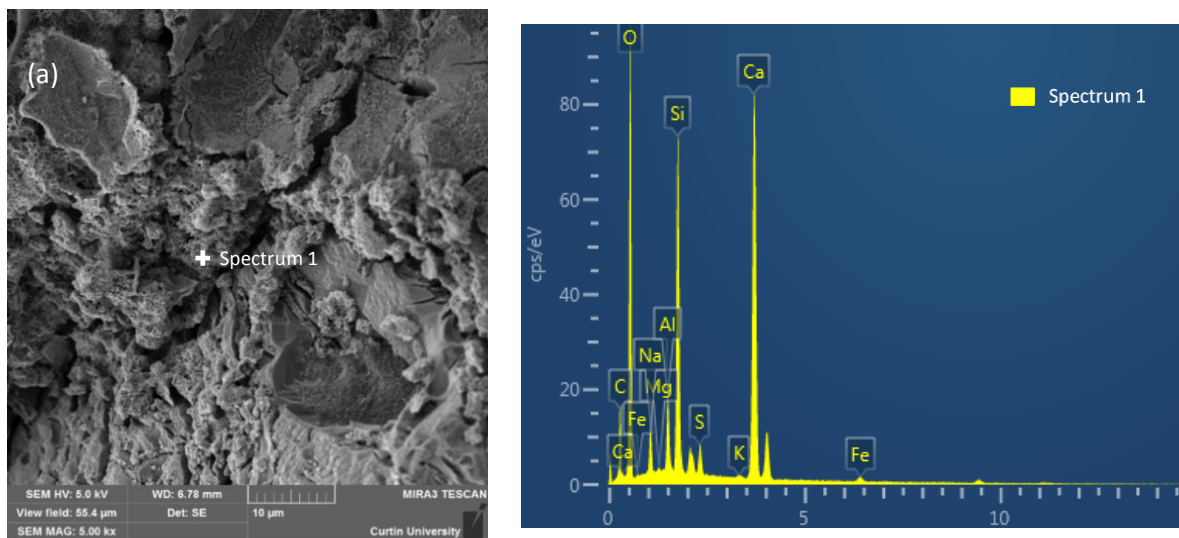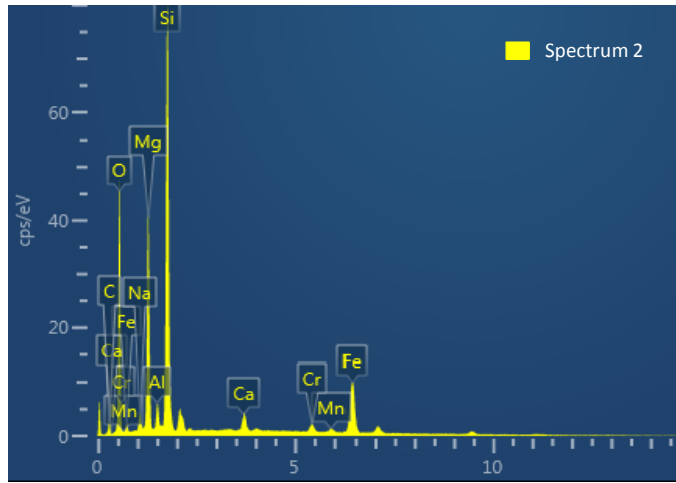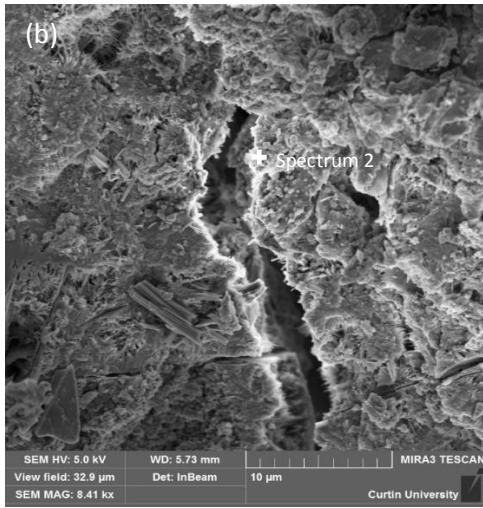
Figure 8. SEM and EDS data of: (a) Mix A1, (b) Mix A5, (c) Mix B1, (d) Mix B5, (e) Mix C1 and (f) Mix C5

The SEM images of the samples containing FNS with 100% OPC (Fig. 8b), and 70% OPC and 30% GGBFS (Fig. 8f) exhibited formation of foil-like crystals and rosette-like accumulation on the aggregate surface and in the micro-cracks. The EDS data of the product in these specimens show that they have high silica content. As a result, they have low Ca/Si ratios as compared to the other specimens. The Ca/Si ratio of the specimens with 100% OPC (A5) and 30% GGBFS (C5) are 1.64 and 1.85 respectively. On the other hand, specimens of the mixtures A1, B1, B5 and C1 have Ca/Si ratios of 2.33, 2.57, 2.59 and 2.44 respectively. The lower Ca/Si ratios of specimens A5 and C5 indicate that the silica of the FNS aggregates

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292 reacted with alkaline pore solution to make the ratio lower. Since there was no FNS aggregate

293 in the mixtures A1, B1 and C1, there was no contribution of silica from the aggregates to the

294 reaction product. Thus, the Ca/Si ratios of these samples were high. Since the specimen B5

295 contained 30% fly ash as cement replacement, there was less Portlandite available for the FNS

296 aggregate to react with. For this reason, the Ca/Si ratio was high for this specimen. Thus, ASR

297 expansion of the specimens using 30% fly ash as supplementary cementitious material was low

298 even though FNS was used as 100% fine aggregate in these specimens.

299

300 **Conclusion**

301   The reactivity of a proprietary ferronickel slag aggregate in cement mortar was studied

302 by using the accelerated mortar bar test. The test consisted of periodic measurement of the

303 length change of mortar specimens while immersed in 1M NaOH solution at 80 ºC. The FNS

304 aggregate was classified as reactive according to the Australian Standard AS 1141.60.1. Uses

305 of class F fly ash and GGBFS as supplementary cementitious materials were investigated as

306 the ASR mitigating measure of the FNS. It was found from the AMBT results that 30% fly ash

307 reduced the 21-day expansion of mortar bars containing up to 100% FNS to below 0.3% limit

308 set by the Australian Standard. The expansions of the other specimens containing OPC alone

309 or with 30% GGBFS in the binder and FNS content of 25% or more were above this limit. The

310 expansions of these specimens increased significantly for continued immersion up to 64 days.

311 The expansions of the specimens with 30% fly ash and up to 100% FNS remained below 0.3%

312 for 64 days of immersion. The SEM photographs and EDS data showed the presence of ASR

313 products on the surface of FNS aggregates in the specimens except those using 30% fly ash.

314 Thus, 30% fly ash as a supplementary cementitious material was found as an effective measure

315 for mitigating the ASR expansion of the ferronickel slag aggregate up to 100% replacement of

316 natural sand.

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