

# ALKALI SILICA REACTION OF RECYCLED GLASS AGGREGATE MORTAR USING FLY ASH UNDER STEAM CURING

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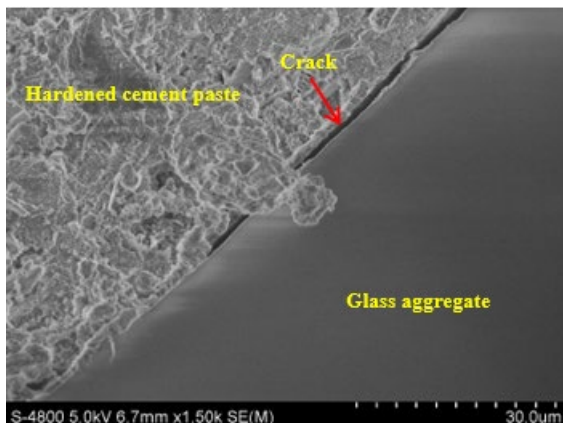
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## Graphical abstract



## Abstract

Utilization of recycled glass as fine and coarse aggregates in concrete materials can give significant economical and environmental benefits, especially in the case of developing countries. However, alkali-silica reaction (ASR) will be a serious problem when the recycled glass is used as concrete aggregates. This paper shows an investigation of the alkali-silica reaction of glass sand mortar under steam curing. The test for ASR expansion follows ASTM C1567. Type F fly ash (FA) is used together with Ordinary Portland cement (OPC) mixing with mixed color glass sand. FA contents are of 20%, 30%, 40% and 50% as OPC replacement. Steam curing accelerating FA reaction is used to contribute to the mitigation of ASR by strength improvement of the mortar. Under steam curing, the results show that the recommended contents of FA to use are of 20% to 30% to ensure expansion lower than the threshold of ASTM. Moreover, steam curing increases the expansion of the glass sand mortar in compared to the moist curing, and expansion crack appears at the interface between glass sand and cementitious matrix, not in the glass sand. In comparison with glass sand mortar under moist curing, expansion of glass sand mortar using fly ash under steam curing is contributed primarily by: (1) delayed ettringite formation, (2) reducing ion transportation, (3) increasing tensile strength of fly ash glass sand mortar and (4) reducing glass sand dissolution rate in rich alkalinity solution.

**Keywords:** Recycled glass aggregate, alkali-silica reaction, steam curing, fly ash.

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## 1.0 INTRODUCTION

At present, developing countries in ASEAN are facing municipal solid waste (MSW) including Vietnam and Thailand. Generally, the MSW of Vietnam is around 0.7kg/person/day, whereas it is of 1.0kg/person/day in the case of Thailand. We have the same serious problem needed to solve is lacking a good MSW management system which includes the collection, treatment, and recycling. Most MSW is exhausted to the environment as open dumps, landfills, and incineration. As a result, societies of

Vietnam and Thailand are remarkably affected as air pollution, leachate, surface and groundwater contamination, ecological damages, health risk, and socio-economic effect. Normally, in Vietnam, the MSW consists of 26% of waste recycling and 74% waste disposal. Among recycled MSWs, waste glass is highly potential to recycle and reuse as an ingredient in construction material, especially in concrete. Waste glass can be ground into various sizes and used as a replacement of fine and coarse aggregates in the concrete. Every year, Vietnam exhausts the environment around 1,600,000 tons of waste glass. It will be

very useful if the recycled glass aggregate can be applied to replace the whole aggregate in the concrete since it consumes much waste glass for the concrete aggregates.

So far the waste glass has been recycled and used as cullets as aggregates in construction including in pavement layers, in asphalt and concrete mixtures, and as fill material. Application as concrete aggregates can be particularly rewarding since the high production volume of concrete materials can incorporate large quantities of recycled glass. Specifically, in areas with limited availability of durable natural aggregates, recycled glass can be used as a partial aggregate replacement, thus reducing the cost and environmental impact of importing aggregates from elsewhere.

Several previous studies have examined the use of waste glass in concrete. Increasing amounts of waste glass as coarse aggregates in concretes have been reported to decrease the mechanical properties, primarily because of a weak interface [1,2,3,4]. Also, larger particle sizes of glass (greater than 1.2–1.5 mm) are found to facilitate alkali-silica reaction (ASR) in concretes [5]. However, when using ground glass or glass powder in cementitious systems, the particle size is not conducive for ASR to occur, but the potential of high alkali content of glass powder to cause deleterious expansions to need to be accounted for [6].

It shows that the major challenge in using glass aggregates in forms of fine and coarse aggregates in concrete is ASR. Due to high concentrations of amorphous silica in the consumer soda-lime glass (70% by weight), these recycled glass aggregates can react with cement alkalis and produce an ASR gel which, in the presence of moisture, expands and causes cracking and deterioration of concrete [7]. And, regarding ASR, mineral powders including fly ash showed a good ability to mitigate ASR occurring in recycled glass aggregate concrete. Many studies have proposed seven potential mechanisms for mitigation of Alkaline Silica Reaction (ASR) in recycled glass aggregate concrete by fly ash as [8]: alkali dilution, alkali binding, limiting mass transport concrete, improving the tensile strength of concrete, modifying ASR gel, consumption of portlandite and supplying soluble alumina. Among these 7 potential mechanisms, some mechanisms are major to mitigate ASR.

In the case of concrete using fly ash, under standard curing condition, the use of fly ash content from 15% to 25% replacement of OPC reduces significantly ASR [9,10]. Effective content of fly ash to mitigate ASR depends on fly ash type and size of glass aggregate. High CaO fly ash is less effective in mitigating ASR when compared to low CaO fly ash [9,11,12].

Using fly ash, by time, pozzolanic reaction reduces the ion diffusion coefficient as well as water permeability of concrete, as a result, it may reduce ASR swelling due to decreasing water absorption of concrete [13,14]. Moreover, additional pozzolanic C-S-H by using fly ash increases binding alkali and hydroxyl ions which helps to mitigate ASR by removing free alkali and hydroxyl ions of pore solution [15,16]. When ASR is determined by the expansion of mortar bars, due to low reactivity at the early age of fly ash, at 3 first days exposed to NaOH solution, mortar bar containing fly ash has expansion performance which is nearly the same as that of mortar bar consisting of neat cement (OPC). However, over time, the pozzolanic reaction of fly ash increases gradually and produce a large surface of C-S-H to bind alkali ion as well as reduce calcium ion in pore solution through consumption of portlandite. Also, the pozzolanic reaction of fly ash may

perform differently as concrete exposed to steam curing. Under steam curing, hydroxyl ion concentration in pore solution increases significantly due to temperature enhanced hydration, as a result, the pozzolanic reaction of fly ash processes correspondingly to form a large number of C-S-H by a transformation of portlandite at an early age [17,18,19]. It implies that steam curing helps to fasten the pozzolanic reaction of fly ash and may mitigate ASR at an early age right after finishing steam curing. However, after steam curing, concrete will be exposed to further moist curing up to the time of concrete product delivery. Therefore, what will be going on for ASR of concrete containing fly ash after finishing steam curing? Up to now, researches on ASR mitigation of concrete consisting of fly ash and cured in steam curing condition are still limited. Mechanism of fly ash mitigation to ASR in concrete under steam curing is not clear.

This paper shows an investigation of the alkali-silica reaction of glass sand mortar under steam curing. The test for ASR expansion follows ASTM C1567. Type F fly ash (FA) is used together with Ordinary Portland cement (OPC) mixing with mixed color glass sand. FA contents are of 20%, 30%, 40% and 50% as OPC replacement. Steam curing accelerating FA reaction is used to contribute to the strength improvement of the mortar.

## 2.0 MATERIALS AND EXPERIMENTS

Binders including Ordinary portland cement (OPC) and fly ash class F were used. Fine aggregates were natural sand and recycled glass sand. The natural sand had a specific gravity of 2.62 and water absorption of 1.05%, whereas, the mixed color glass sand had the water absorption of 0.22% and a specific gravity of 2.48. The recycled glass sand was mixed color soda-lime glasses which were prepared by crushing washed glass bottles then mixed glass sand consisting of 40% green, 31% amber, and 29% clear glass. The oxide composition of OPC cement, fly ash and mixed color glass was presented in Table 1.

Table 1 Mortar Composition

Oxide	Oxide Composition [%]		
	Cement	Mixed color glass	Fly ash
CaO	63.8	9.22	2.14
SiO <sub>2</sub>	19.82	69.42	52.86
Al <sub>2</sub> O <sub>3</sub>	5.16	1.56	32.14
Fe <sub>2</sub> O <sub>3</sub>	2.94	0.68	5.18
Na <sub>2</sub> O	0.28	13.66	0.64
K <sub>2</sub> O	0.69	0.47	1.47
MgO	2.15	0.72	0.82
MnO	0.11	0.02	0.04
TiO <sub>2</sub>	0.30	0.06	1.45
P <sub>2</sub> O <sub>5</sub>	0.56	0.01	0.24
LOI	1.46	0.41	4.96

Mortar bars and cubic specimens were used to investigate the fly ash capacity of mitigating ASR. Mortar mix proportions were prepared following ASTM C1567. In such a standard, mortars had water to cement ratio (w/c) of 0.47, and 53%

volume fraction of fine aggregate whose size was in the range from 0.15mm to 4.75mm.

Investigated specimens included three groups. First, the standard group which had mortar bars and cubic specimens cast by using OPC and natural sand, these specimens were cured in a moist condition. Second, glass sand group cured in moist condition, mortar consisted of cement, glass sand, and fly ash. Third, the glass sand group cured in steam condition. In the second and the third group of specimens, the fly ash was used to replace cement by weight with various contents as 20%, 30%, 40%, and 50%. Regarding steam curing, all specimens were cast in a steel mold, stood for 4 hours after casting then exposed to steam curing for 6 hours. After finishing steam curing, the specimens were stood for further 2 hours then demolded. The steam curing regime consisted of 3 periods as temperature increase for 2 hours, temperature maintaining for 2 hours, and temperature decrease for the other 2 hours. The steam curing had a maximum temperature of 80°C and 100% humidity. The prepared mortar specimens were used to carry out tests as mentioned below.

### Tests of Compressive and Tensile Strength

Mortar bars of 25x25x250mm prisms and cubes of 50x50x50mm of three specimen groups, after prepared according to ASTM C1567, were used to evaluate tensile and compressive strength respectively. Tests were performed at 5 days submerging specimens in NaOH solution. Regarding ASTM C1567 specimen preparation, specimens were cast, demolded after 24 hours of moist curing, cured in water at 80°C for 24 hours then submerged in a 1M NaOH bath at 80°C. The tensile and compressive strength of the mortar specimens were evaluated following ASTM C348-21 and C109/109M-21 respectively.

### Accelerated Mortar Bar Test

The accelerated mortar bar test was carried out following ASTM C1567 to evaluate the effects of fly ash content and steam curing on reducing mortar bar expansion in comparison with the innocuous threshold of ASTM. Three specimen groups were tested with 7 mixtures. With each mortar mixture, four 25x25x250mm prism specimens were cast in steel mold consisting of embedded stainless gauge studs at the opposite ends. Expansion through length measurement of the mortar bar was recorded by using a digital comparator with an accuracy of 0.0025mm. After 24h under moist curing, the mortar bars were demolded and measured initial lengths. Similarly, prism lengths were recorded after 24h of hot water curing at 80°C, and 1, 3, 5, 7, 10, and 14 days submerged in the NaOH bath at 80°C. NaOH bath was sealed to prevent water evaporation. The variation of accelerated mortar bar test was controlled within limitation of 5%.

### SEM/EDS Imaging

SEM/EDS imaging was used to study ASR formation, ASR composition, and mortar microstructure, especially microstructure at the interface between glass sand and cementitious matrix. After ASTM C1567 test, cross-sections were cut with a diamond blade saw from prisms with an approximate thickness of 1cm. The SEM/EDS imaging was

conducted with 100% glass sand mortar bar cured in moist condition, 100% glass sand mortar bar cured in steam condition and 100% glass sand mortar bar consisting of 20% fly ash steam cured. SEM was equipped with an X-ray energy dispersive spectroscopy (EDS) detector to analyze ASR gel composition.

## 3.0 RESULTS AND DISCUSSION

### Effect of Fly Ash on ASR Expansion under Steam Curing

The expansion of mortar bars cast with mixtures of three groups following ASTM C1567 is shown in Figure 1. In this figure, standard specimen series is mortar consisting cement and natural sand and moist cured, control-S is mortar casted using cement – glass sand and steam cured, whereas control-M is mortar using cement-glass sand and moist cured, 20F-S up to 50F-S is mortar prepared with 20% to 50% fly ash replacing cement together with glass sand and steam cured, while 20F-M up to 50F-M is mortar having 20% to 50% fly ash replacement of cement mixed with glass sand and moist cured. Results are shown in Figure 1 propose that fly ash F class used to replace cement can effectively mitigate ASR with content both of 20% or higher. Both in steam curing or moist curing, at a specific time submerged in NaOH 1M solution, increasing fly ash content makes the expansion of the mortar bar smaller when compared to that of the control mortar bar. Also, to mitigate ASR expansion below the threshold of ASTM, 20% fly ash is a sufficient content recommended to use in the mortar bars, however, under the steam curing, to ensure high confidence in ASR mitigation, a higher fly ash content than 20% should be considered (i.e., 30% fly ash). All mortar bars show very small expansion or so-called negligible in the first 3 days exposed to NaOH. From the 3 days to 5 days, the expansion of control specimens started gradually and became clear at 5 days, especially, beyond 5 days exposure, the expansion of control mortar bar increased drastically and likely constant. In the case of using fly ash, after 3 days of exposure to NaOH 1M, the expansions still showed slowly increasing up to a conclusion of the ASTM C1567 test which was lower than the threshold of ASTM. Phenomena of negligible expansion of the mortar bars at the first 3 days imply that it takes at least 3 days for OH<sup>-</sup> ion to penetrate from NaOH bath into insight the mortar bar and reach a critical concentration to make ASR initiation. This result agrees with other researches of ASR mitigation by using fly ash [8,9].

Figure 2 shows a comparison of the expansion of mortar bars cured in steam and moist condition, in which, mortars were prepared with 20% fly ash and glass sand. After 3 days, the expansion rate of the steam cured glass sand mortar bar was higher than that of moist curing. At a specific time of exposure to NaOH solution, the expansion of the mortar bar cured in steam condition was always higher than cured in a moist condition. The expansion of glass sand mortar steam cured at the conclusion following ASTM C1567, although which was lower than the threshold of ASTM, was higher 3 times when compared to that in moist curing. Two reasons are supporting to the higher expansion of the steam cured glass sand mortar, they are: (1) ettringite delayed formation due to steam curing at 80°C at 4 hours right after casting mortar bar. At a hypothesized temperature over 70°C, ettringite is not formed, however, after demolding and exposed to NaOH solution, the

temperature in mortar bar goes down and favors ettringite formation again which results in a contribution to increasing expansion of the mortar bar, the ettringite formation was recorded by SEM imaging analysis section. The delayed ettringite formation appears in both control and fly ash mortar bar cured in a moist condition also [9]. However, in steam curing, the delayed ettringite formation occurs as soon as the mortar bar exposed to high temperatures at the early hours. (2) porous interlayer CSH favors OH<sup>-</sup> ion transportation from NaOH bath into the mortar bar, as a result, ASR gel forms due to sufficient OH<sup>-</sup> concentration in pore liquid. Some other studies reported that under steam curing, portlandite formation is drastically transformed to CSH with the presence of fly ash, however, due to distribution of a large number of fine pore located at interlayer among CSH layers, this CSH type becomes more porous when compared to CSH microstructures of mortar cured in moist condition [17,20].

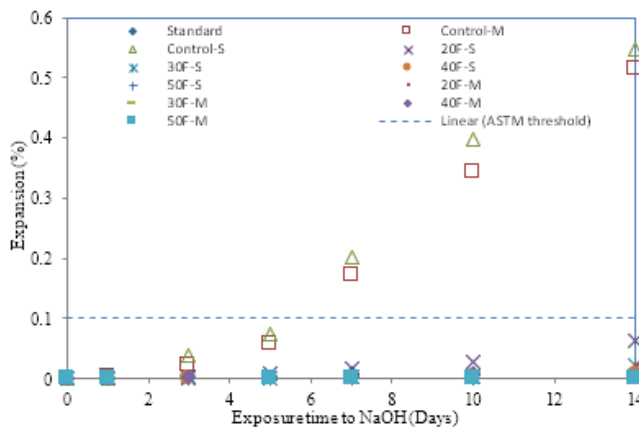


Figure 1 ASR expansion of mortar bars prepared with three specimen groups

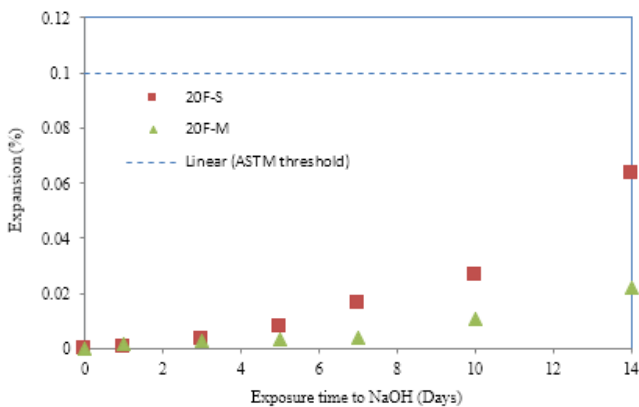


Figure 2 ASR expansion of mortar bars prepared with 20% fly ash cured in steam and moist condition

Typically, in steam curing, increasing fly ash content of replacing cement mitigated effectively ASR as shown in Figure 3. After ASTM C1567, the expansions of glass sand mortar bars were much lower than the threshold value (0.1%) by ASTM, especially with using 30% up to 50% fly ash. Fly ash increase contributes to the alkalinity of pore solution throughout CH consumption and enhancement of alkali binding on the hydrate surface. Besides, fly ash content increasing gives a large silicate surface area and reduces significantly the ratio of OH<sup>-</sup> to silicate

surface area [9], this reduced ratio prevents glass sand dissolution in a high OH<sup>-</sup> concentration of pore solution due to fly ash dissolution performed by hydroxyl ions.

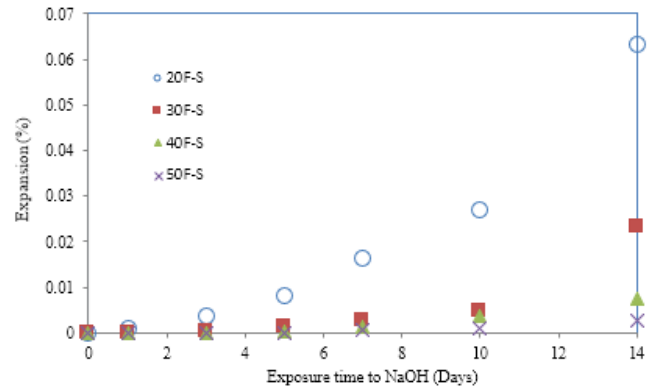


Figure 3 Effect of fly ash content on ASR expansion of mortars cured in steam condition

### Tensile and Compressive Strength of Mortar

Standard mortar and glass sand mortar were tested compressive and tensile strength after 5 days exposure to NaOH 1M as ASR became readily for 5 days. The results of the compressive strength of the mortars are shown in Figure 4. It presents that all steam cured glass sand mortar had lower compressive strength when compared to the compressive strength of the standard mortar. It was very interesting that glass sand mortar using 20% up to 50% fly ash had higher compressive strength than the control mortar cured in steam condition. Using 20% of fly ash increased by about 56% compressive strength compared to the control specimen with 100% cement. Even with 50% fly ash replacing cement, the mortar still had a 38% increasing of compressive strength. The tensile strength has a proportional relationship with compressive strength, therefore, using the fly ash content also increased the tensile strength of the glass sand mortar cured in the steam condition, this increase is shown in Figure 5. Increasing the tensile strength of the mortar using fly ash was nearly 60% and 50% with 20% and 50% fly ash respectively. Generally, due to the expansion of ASR formation, micro-cracks may occur at the interface between cement matrix and glass sand or inside glass sand and decreases the strength of the mortar. However, in the case of using fly ash and steam curing, those micro-cracks and pores will be densified and contribute to an enhancement in the strength. Seyed [9] reported that the improvement of the tensile strength of mortar helps to prevent micro cracks and results in mitigating ASR by slowing down NaOH solution penetration through the cracks and allow the further hydration of the binder to densify cementitious matrix and reduce ion transportation. It is obvious to propose that the steam curing increased significantly the tensile strength of the mortar at an early age which contributes primarily to ASR mitigation.



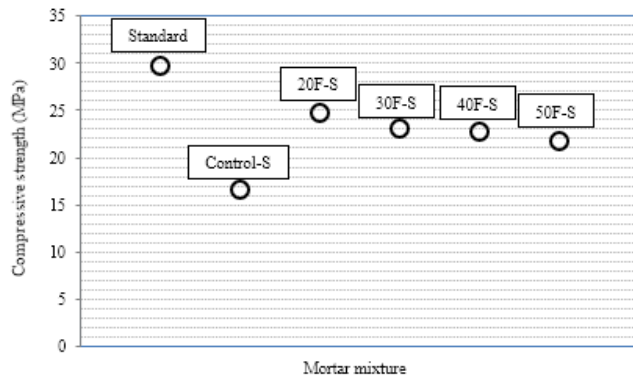


Figure 4 Compressive strengths of mortars (cured in steam condition) 5 days after submersion in 1M NaOH solution at 80°C

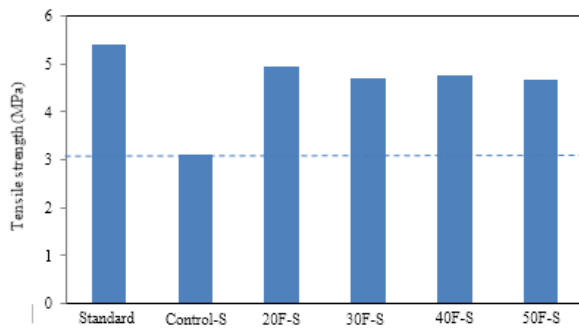
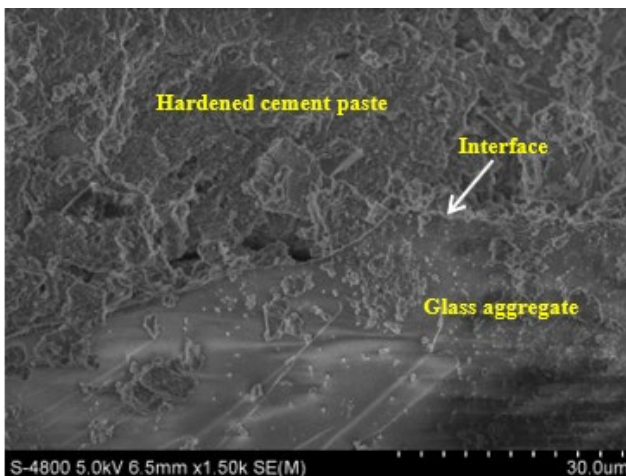


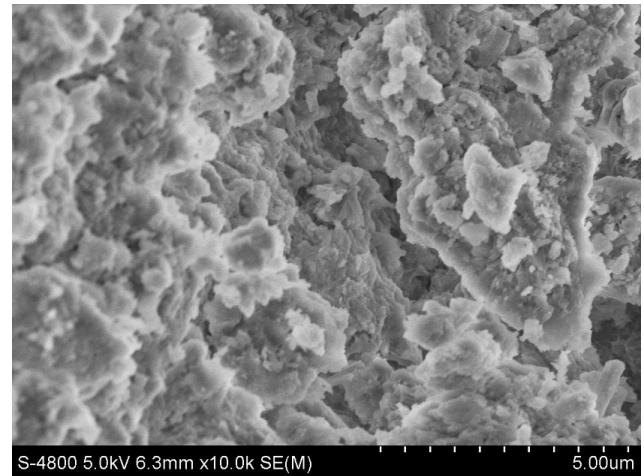
Figure 5 Tensile strengths of mortars (cured in steam condition) 5 days after submersion in 1 M NaOH solution at 80°C

SEM/EDS Analysis of Mortar

After 14 days of exposure to 1M NaOH, mortar bars were evaluated microstructure by SEM imaging and analyzed the composition of ASR gel formed. SEM micrographs of 20F-S and control-S mortar bars are shown in Figures 6 and Figure 7 respectively. SEM micrographs of 20F-S mortar bars proved that there was no crack at the interface and CSH microstructure became more porous under steam curing. The interface microstructure was densified.



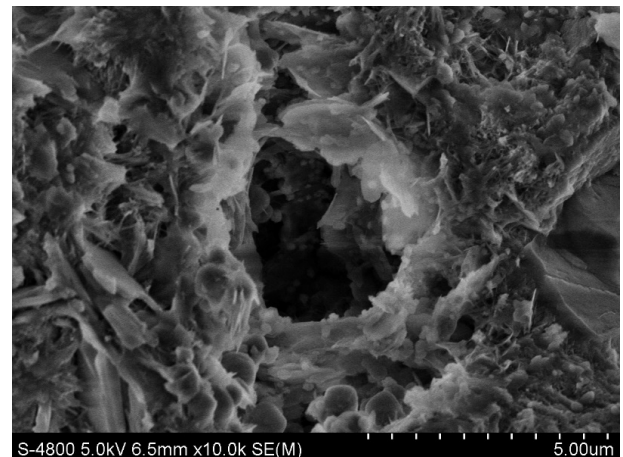
(a)



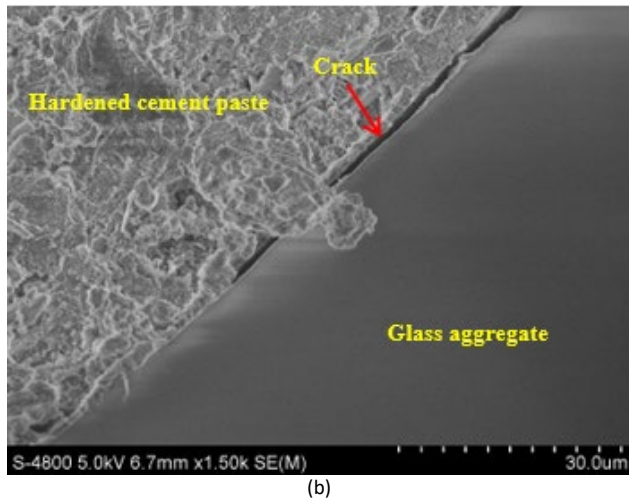
(b)

Figure 6 SEM micrographs of 20F-S mortar bar at 14 days after submersion in 1 M NaOH solution at 80°C: (a) interface between glass sand and cementitious matrix ; (b) microstructure of CSH in the matrix

Especially, under steam curing, SEM micrographs of control-S mortar bars showed very clearly a crack appearance along the glass sand side. The interface microstructure was not densified. Also, many hydrates were appearing simultaneously in the matrix surrounding the glass sand, these hydrates were CH, CSH, Afm, and Aft. Moreover, Figures 6 and Figure 7 do not show crack appearance in glass sand, this result is different from reports of Farshad [5] and Seyed [9] whose results showed cracks in glass sand. Crack appearance at the interface of control-S mortar bars provided that the steam curing may result in a sufficient expansion due to delayed ettringite formation to initiate a crack along the glass sand side. With using fly ash combined steam curing, the crack was densified by CSH then tensile strength of the mortar bar was improved to make ASR expansion be mitigated.



(a)



**Figure 7** SEM micrographs of Control-S mortar bar at 14 days after submersion in 1 M NaOH solution at 80°C: (a) hydrates appearance near the interface; (b) crack at interface between glass sand and cementitious matrix

EDS composition analysis of ASR gels of control mortar cured in moist, in steam, and mortar consisting of 20% fly ash moist cured and steam cured is presented in Table 2. As shown in Table 2, there is no clear difference among composition analyses of these mortar bars in terms of Ca/Si and Ca/Na. Results showed evidence that using fly ash reduced Ca/Si and that the steam curing made Ca/Si increase. This implies that the control mortar bars have more ASR gel formed which initiates cracks or more expansion, additionally, because of higher Ca/Si in ASR gel, the control mortar cured in steam condition has a superior expansion to the control mortar under moist curing. The higher Ca/Si in ASR gel proved that pore solution of the mortar may present a higher alkalis concentration which promotes a reaction between amorphous silica in the consumer soda-lime glass in recycled glass aggregates with cement alkalis to provide an ASR gel that a superior expansion of the control mortar bar appears.

**Table 2** Average Atomic of ASR from Mortar Bars Measured by EDS after 14 Days Exposure to NaOH 1M (By Weight, %)

Mix series	Ca	Si	Na	K	Al	Ca/Si	Ca/Na
Control-S	6.98	22.69	11.16	0.38	0.88	0.31	0.63
Control-M	6.52	23.12	10.93	0.42	0.81	0.28	0.60
20F-S	5.36	25.84	6.62	0.78	1.06	0.21	0.81
20F-M	4.96	26.91	6.15	0.92	0.97	0.18	0.81

#### 4.0 CONCLUSION

Results of this investigation on the effectiveness of the fly ash F type on mitigation of ASR of glass sand mortar under steam curing propose the following conclusions:

- Under steam curing, according to the test of ASTM C1567, 20% to 30% fly ash replacing cement is sufficient to mitigate ASR expansion of the glass mortar. It is recommended to use 30% fly ash to ensure expansion lower than the threshold of ASTM.

- Steam curing increases the expansion of the glass sand mortar in compared to the moist curing. And, after ASTM C1567 test, expansion crack appears at the interface between glass sand and cementitious matrix, not in the glass sand.
- In comparison with glass sand mortar under moist curing, expansion of glass sand mortar using fly ash under steam curing is contributed primarily by: (1) delayed ettringite formation, (2) reducing ion transportation, (3) increasing tensile strength of fly ash glass sand mortar and (4) reducing glass sand dissolution rate in rich alkalinity solution.

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