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Using lithium nitrate and pozzolanic glass powder in concrete as ASR suppressors

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Abstract

This investigation studies the influence of two different mineral admixtures, lithium nitrate (Li) and pozzolanic glass powder (PGP) on the expansion induced by alkali-silica reaction (ASR). Four numbers of concrete prisms were produced for each concrete mix to measure the expansion resulted from the ASR according to the test method of the BS 812-123:1999. Chemical analysis was performed using X-ray spectra. Test results confirmed that Li and PGP have significantly reduced the ASR expansion. Lower calcium to silica ratio (Ca/Si) was found in concrete mix contains (PGP) because of the high amorphous reactive silica and low calcium content in PGP compared to ordinary Portland cement CEM1. Similar components and minerals phases were obtained in different concrete mixes by using XRD.

Keywords: Pozzolanic glass powder; Lithium nitrate; Recycled glass sand; Alkali-silica reaction; ASR suppressors; SEM; XRF; XRD

1. Introduction

Alkali-silica reaction (ASR) is a deleterious reaction occurring between reactive siliceous minerals present in some aggregate and the alkaline pore solution in concrete. Since the problem of the ASR expansion was voiced, numerous research works were carried out worldwide to eliminate the deterioration effect of ASR.

The use of pozzolanic glass powder is newly introduced as a novel method to mitigate ASR expansion. The amorphous structure of the glass promotes it to undergo pozzolanic reaction with the presence of alkaline activator, such as: lime, cement and alkalis (sodium and potassium) and contribute to the C–S–H structure and hydration products.

For more than 50 years, researchers have recognized that lithium compounds can effectively prevent the expansion caused by ASR, and in recent years there has been renewed interest in using lithium compounds either as an admixture in new concrete.

In this study pozzolanic glass powder and lithium nitrate were used in concrete as cement replacement and chemical admixture respectively to mitigate the expected ASR expansion in concrete prisms due to the presence of waste recycled glass particles in the concrete as sand replacement.

2. Using recycled glass in concrete

The earliest research work that studied the potential risk of ASR expansion in concrete with the presence of recycled glass as aggregate replacement was carried out by Schmidt and Asia [1], while the first research work that investigated the possibility of utilising very fine grounded powdered glass as pozzolanic material was completed in 1973 [2]. Nevertheless, currently the percentage of utilisation waste recycled glass in concrete is not at a desired level, because of the potential ASR risk and lack in the information and outcomes of using waste recycled glass in concrete, in particular, the long term observations.

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3. Chemistry and mechanism of ASR

The ASR starts when the high alkaline pore solution of the Portlandite attacks the siliceous minerals in the reactive and unstable aggregate. The product of this chemical reaction is known as the alkali-silica gel which capable of absorbing water and swell with a consequent tendency to increase in volume and generate internal pressure. The alkali-silicate gel is formed, either in planes of weakness or pores in aggregate or on the surface of the aggregate particles. In the latter case, a characteristic altered surface zone is formed which might be able to destroy the bond between the aggregate and the surrounding hydrated cement paste, particularly in a humid environment. As the alkali-silicate gel is confined by the surrounding hydrated cement paste, the produced internal pressure may eventually lead to expansion, cracking and disruption of the hydrated cement paste by severe damage to the whole concrete structure [3,4].

3.1. Minimising ASR by low calcium ashes

Chatterji [5], Diamond [6] and Helmuth [7] believed that the alkali-silicate gel formation takes place only in the presence of enough amount of Calcium ions (Ca^{++}) in concrete. Therefore, even in concrete with very high alkali (Na_2O_{eq}) content, if the cement or any other cementing binder that used in the concrete has low calcium content, the ASR expansion will be mitigated. This phenomenon can be explained as follows; the silica in the reactive aggregate will simply dissolve in alkali-hydroxide solution, and will not be available to form any alkali-silicate gel. This is of importance with reference to the prevention of the expansive ASR by the inclusion of Pozzolanic powders of low calcium content such as GGBS, PFA and Metakaolin to replace the cement in the concrete [8].

3.2. Minimising ASR by lithium compounds

Lithium is recognized as a soft, silver-white, and lightest dense metal and has an atomic number of three (3), with a density about half that of water (0.53 g/cm^3). It is a very reactive metal, because of its tendency to expel its outer electron. Lithium (Li) is classified as an alkali metal [9].

McCoy and Caldwell [10] were the first who studied the possibility of using lithium salts to mitigate the ASR. Thereafter, various lithium compounds were investigated over many years to evaluate their effectiveness in mitigating alkali-silica reactivity [11–16].

It has been found in previous research works that more lithium is taken up by the hydrated cement to form the hydration products rather than the sodium and potassium when Lithium compounds were used in concrete. The ameliorative results produced by lithium appears to involve the incorporation of lithium into the production of the ASR gel, and the presence of lithium in the structure of the ASR gel will reduce the tendency of the ASR gel to absorb water and swell.

It was agreed in the previous studies that the greater proportion of lithium as against other alkali components in the concrete mix incorporated in the alkali-silica gel will reduce the chance of the destructive expansion taking place. Therefore, a high lithium dosage will be necessary to get sufficient amount of the lithium that taken up by the ASR gel. Therefore, it can overcome the concomitant increase in the OH^- ion challenge [17].

4. Experimental programme

The experimental programme in this study was divided into the following:

- Identify the chemistry of the cementitious materials and sand which were used as ingredients of concrete mixes.
- Find out the alkali content of the ordinary Portland cement CEM1 and pozzolanic glass powder PGP.
- Secure enough amount of reactive silica in the concrete mix to induce the alkali-silica reaction by using waste recycled glass particles as sand replacement.
- Prepare concrete prisms for ASR test in accordance with test method of the BS 812-123:1999.

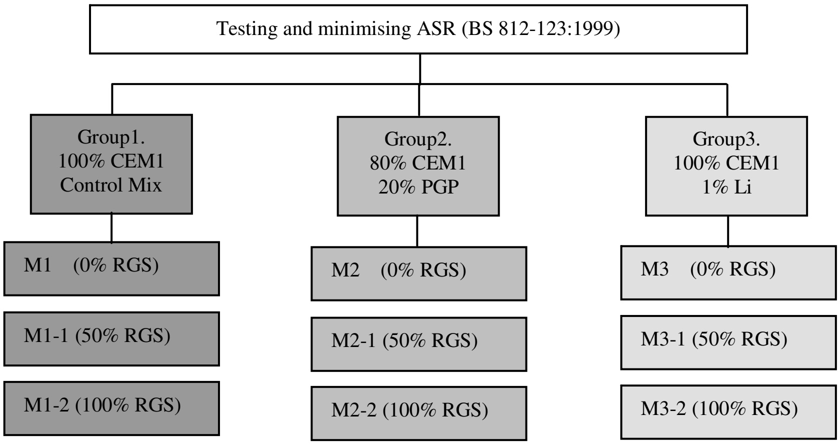


Fig. 1. Testing and minimising programme for ASR expansion.

- Utilize 20% of pozzolanic glass powder as cement replacement, and 1% of lithium nitrate as chemical admixture in concrete mixes in order to mitigate the ASR.
- Explore the chemistry of the concrete by using X-Ray Fluorescent and X-Ray Diffraction.

5. BS 812 part 123:1999 test method

The potential risk of the alkali-silica reaction in concrete was monitored in accordance with the British Standard method BS 812 part 123:1999 [18]. The test uses four concrete prisms with cross-section of 75 × 75 × 279 mm prisms. These prisms are cured for 7 days at room temperature whilst held in cylinders that maintain a high humidity. After curing, the cylinders are kept into an environmental chamber with controlled temperature of 38 ± 2 °C. The measurements of prisms length are made at ages of 1, 2, 4, 13, 26, 39 and 52 weeks using length comparator apparatus.

The alkali content in the concrete mix represented in the amount of the sodium equivalent Na_2O_{eq} was adjusted in accordance with the BS 812 part 123:1999, such that it is comparable to that of mix prepared using cement having Na_2O_{eq} of $1.00 \pm 0.05\%$. This was done by adding (if required) of an appropriate quantity of potassium sulphate K_2SO_4 to the mixing water [18]. Fig. 1 shows the testing and minimising programme for ASR expansion.

6. Materials

The materials used in this study were

Portland cement: Ordinary Portland Cement (CEM1) had a strength class 42.5 N according to the British standard BS EN 197 Part 1:2000 [19]. The alkali content (Na_2O_{eq}) of the CEM1 was 0.58%.

Pozzolanic glass powder (PGP): Very fine pozzolanic glass powder (PGP) with an average size particle of sub 45 μ was used in this study as cement replacement. The alkali content (Na_2O_{eq}) of the PGP was 14.16%.

Lithium nitrate $LiNO_3$(Li): Anhydrous lithium nitrate salt $LiNO_3$ that specified as a reagent for general laboratory work was used through out this study supplied by Fisher Scientific [20].

Natural aggregate: Natural limestone crushed rock and Sea Dredge Sand (SDS) were used throughout this study as coarse and fine aggregate.

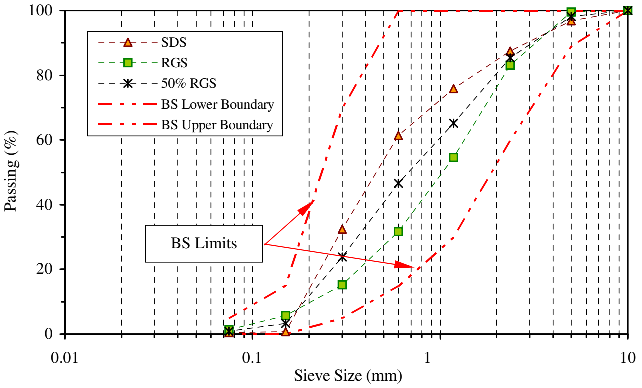


Fig. 2. Sieve size analysis for SDS and RGS.

Table 1
Relative density and water absorption for cementitious materials and aggregates

Material		Relative density (ton/m^3)		Water absorption (%)
		OD[a]	SSD[b]	
Cement	CEM1	3.14	–	–
	PGP	2.50	–	–
Coarse aggregate (crushed limestone)	20 mm	2.66	2.67	0.60
	10 mm	2.66	2.68	0.66
Fine aggregate (Sand)	Sea dredge	2.60	2.63	1.00
	Recycled glass	2.50	2.51	0.06

[a] OD: is the oven dry density.
[b] SSD: is the saturated surface dry density.

Recycled glass sand (RGS): Recycled glass sand (RGS) of particle size less than 5 mm, supplied by Day Aggregate Ltd. The results of the sieve analysis, relative density and water absorption for sea dredged sand (SDS) and recycled glass sand (RGS) are available in Fig. 2 and Table 1, respectively.

The chemical analysis for the major oxides of the cementitious materials (CEM1, and PGP) and the sands (SDS and RGS) used in this study obtained by the XRF are available in Table 2. The content of the Calcium Ca and Silica Si in the raw materials is shown in Fig. 3. The alkali content of CEM1 and PGP is shown in Table 3.

Table 2
Chemical composition of the cementitious materials and sands

Materials		CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O	TiO_2	Mn_2O_3	SrO	P_2O_5	Cr_2O_3
Cement	CEM1	64.5	20.4	5.63	2.85	1.09	0.18	0.64	0.27	0.06	0.09	0.16	<0.05
	PGP	8.61	72.3	1.04	0.17	3.89	13.31	0.52	<0.05	<0.05	<0.05	<0.05	<0.05
Sand	SDS	7.11	78.6	2.55	2.47	0.46	0.42	0.64	0.15	0.10	<0.05	0.09	<0.05
	RGS	10.63	72.1	1.78	0.36	1.26	12.40	0.64	0.06	<0.05	<0.05	<0.05	0.09

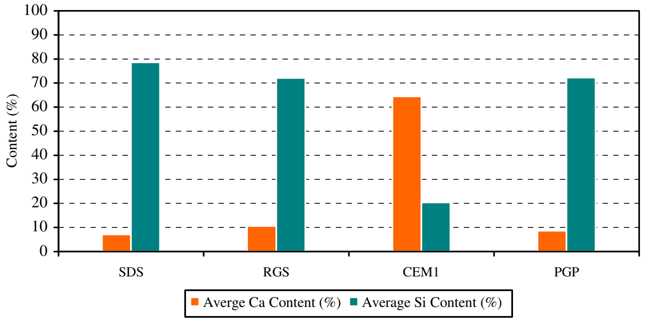


Fig. 3. Average content of Ca and Si in the raw materials.

Table 3
Alkali content in the cementitious materials

Cement type	Na_2O	K_2O	Na_2O_{eq}
CEM1	0.16	0.64	0.58
PGP	13.82	0.52	14.16

7. Mix proportions

Two levels of replacement for the recycled glass sand to replace the natural sand were used in this study, namely 50% and 100%, while single replacement of 20% was used for the PGP to replace the CEM1. Three different groups of concrete mixes were prepared. Each group was subdivided into three concrete mixes (Table 4).

The BS 812 Part 123:1999 specifies proportions for the ASR test concrete mix (Table 5). The proportions are expressed in terms of volume. Water absorption of the aggregates and moisture correction are also taken into consideration to achieve constant free water content for all the mixes. Four prisms were produced from each mix for testing.

8. Alkali-silica reaction test

In this study, the potential risk of alkali-silica reaction that can be occurred due to the use of waste recycled glass as sand replacements at levels of 50% and 100% by mass was investigated. The alkaline content of the concrete described as sodium equivalent Na_2Oeq content in the three groups of concrete was 4.07, 23.08 and 4.07 kg/m^3 for group 1, 2 and 3, respectively. In order to maintain the alkaline content in the concrete mix to 1.00 ± 0.05%, potassium sulphate K_2SO_4 was added to mixing water (if required). The required mass of potassium sulphate to be added for each concrete mix was calculated according to BS 812-123:1999 as shown in Table 6 below.

Table 4
Concrete mixes groups and subgroups

Group ID		1-Control	2-PGP	3-Li
Binder type		100% CEM1	80% CEM1 20% PGP	100% CEM1 1% $LiNO_3$
RGS content	0%	M1	M2	M3
	50%	M1-1	M2-1	M3-1
	100%	M1-2	M2-2	M3-2

Table 5
Concrete mix proportions for ASR test

Cement	Water	>20 mm	<10 mm	Sand
22.2%	22.8%	16.5%	22.0%	16.5%

8.1. The use of pozzolanic glass powder (PGP) as cement replacement

Glass powder as a pozzolanic amorphous material when it is used in concrete as cement replacement can contribute to the formation of the hydration products and the C–S–H gel. Moreover, the results of this study showed that when PGP was used in concrete as cement replacement, the ASR expansion was significantly reduced even with high alkali content. The possible explanations for this phenomenon are discussed below:

- It is believed that due to the contribution of the PGP in the hydration process and chemical reaction, the chemistry, concentration and nature of the pore solution and OH^- ions were experienced changes that lead to mitigate the ASR.
- The available reactive silica in PGP as an amorphous material will dissolve very quickly during the pozzolanic reaction and react with other chemicals to form the mineral phases of concrete. Therefore, the dissolved reactive silica of PGP will be accommodated in the crystals of concrete minerals and not available for the ASR which occurs in later stages. It was concluded that, with the presence of other chemicals the dissolved reactive silica of PGP will not be able to form ASR gel during the pozzolanic reaction, or even a gel having a swelling property likewise the ASR gel. Otherwise, if there is any chance to form ASR gel during the pozzolanic reaction from the reactive silica of PGP one can envisage that any existing ASR expansion will be noticeable. Another conclusion stated that if any ASR can be occurred during pozzolanic reaction stage, the produced expansion would vanished and accommodated with the plasticity of fresh concrete.

Table 6
Alkali content and the required K_2SO_4 according to the BS 812-123:1999

Group ID	1	2	3
CEM1 content	100%	80%	100%
Mineral additives	0	20% PGP	0
Na_2O eq. (%)	0.58	3.30	0.58
Alkaline content (kg/m^3)	4.07	23.08	4.07
Added K_2SO_4 (kg/m^3)	8.25	*n/a*[a]	8.25

[a] Very high alkaline content in the concrete mix, above 1%, because of the contribution of the PGP.

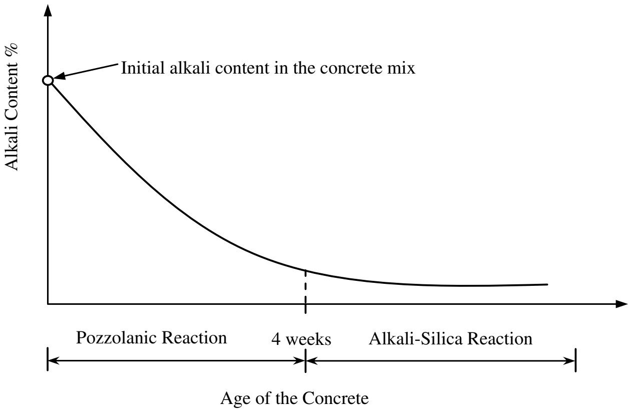


Fig. 4. Model illustrates the consumption of alkali in the pozzolanic reaction in the first 4 weeks.

- Due to the lower calcium content in the PGP compared to CEM1, the amount of Portlandite [$Ca(OH)_2$] produced in concrete contains a mixture of CEM1 and PGP was lower than that of concrete contains only CEM1. Therefore, the concrete was considered of a mild reactivity and the risk of ASR was reduced.
- In the first 4 weeks of the pozzolanic reaction and hydration process, most of the alkalis were consumed in order to catalyst the pozzolanic reaction and act as chemical activators to compensate the lower calcium content in the PGP. Therefore, there was not be enough free alkali to interact with the reactive silica in the pore solution of the concrete to induce ASR when it's due in later age as illustrated in Fig. 4.

8.2. Discussion

The presence of the recycled glass sand in the concrete mix possesses high deterioration risk of ASR cracking and deterioration as shown in Fig. 5. Concrete mixes M1-1 and M1-2 experienced severe ASR cracking. Fig. 6 shows the expansion of ASR results of concrete mixes M1, M1-1 and M1-2 for the period of 52 and 78 weeks.

Fig. 7 reveals the effect of the PGP on mitigating ASR expansion of the concrete mixes M2, M2-1 and M2-2 in which 20% of the CEM1 was replaced by PGP. The effect of using lithium nitrate in concrete mixes of group 3 on ASR expansion is represented in Fig. 8.

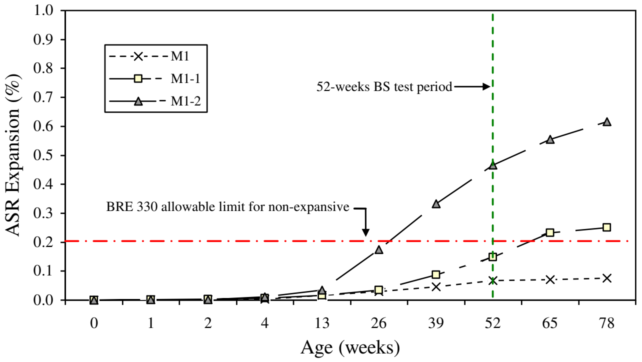


Fig. 6. ASR expansion for concrete mixes of group 1.

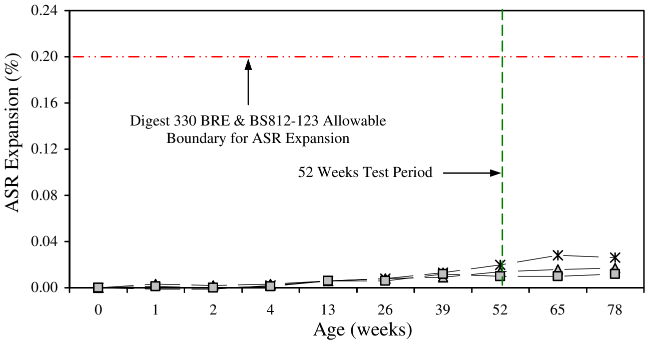


Fig. 7. ASR expansion for concrete mixes of group 2.

Fig. 9 illustrates the behaviour of normal concrete where no recycled glass sand was used in the concrete mixes. In case of no recycled glass sand was in concrete mixes, all of the concrete mixes of the three groups did not exhibit any expansion as the ASR expansion did not exceed the allowable limits stated by the BRE Digest 330 [21] and the BS 812-123:1999.

A noticeable expansion was monitored in mix M1-1 when 50% of the primary sand was replaced by the recycled glass sand, although the expansion was below the allowable limit for non-expansive aggregate for 52 weeks, but the case can be considered as a potential risk as the ASR expansion was increased with time as shown in results taken at the period of 78 weeks (Fig. 10). When 100% of the primary sand SDS was replaced by recycled glass sand RGS, the

M1 (0% RGS) M1-1 (50% RGS) M1-2 (100% RGS)

Fig. 5. ASR cracks in concrete mixes – group 1. No ASR suppressors were used.

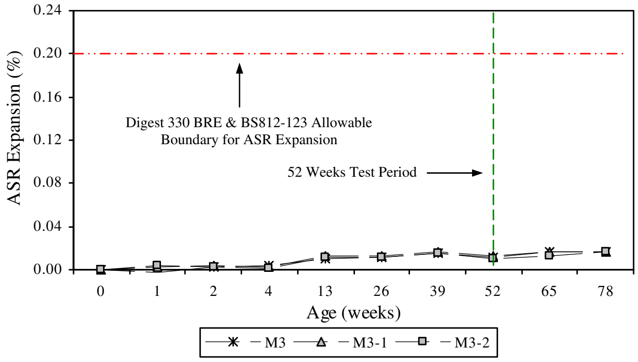


Fig. 8. ASR expansion for concrete mixes of group 3.

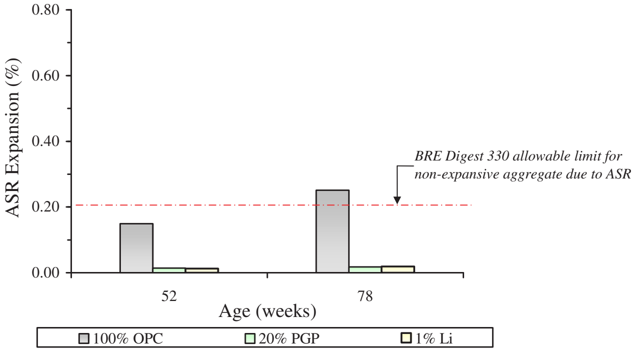


Fig. 10. ASR expansion at 52 and 78 weeks, 50% RGS.

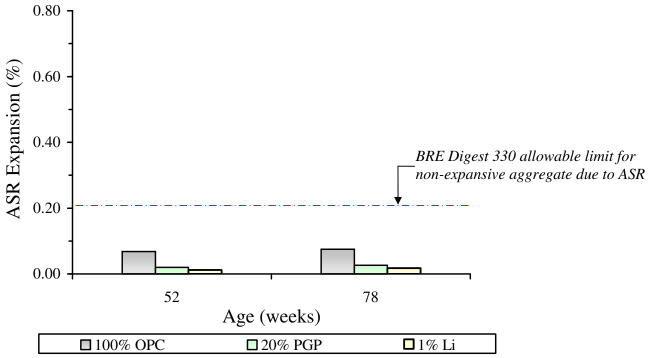


Fig. 9. ASR expansion at 52 and 78 weeks, 0% RGS.

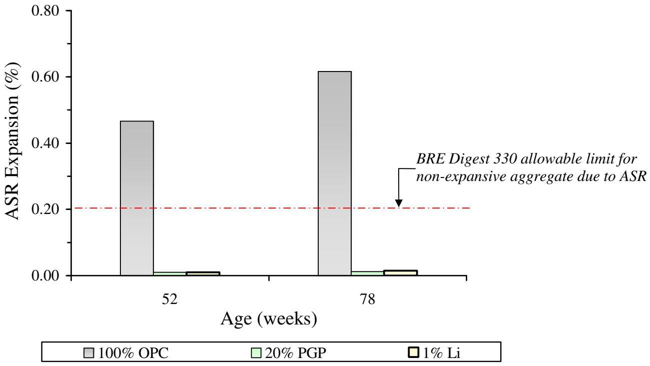


Fig. 11. ASR expansion at 52 and 78 weeks, 100% RGS.

expansion of the ASR was very high compared to the allowable limit of the non expansive aggregates (Fig. 11). Nevertheless, all of the ASR suppressors have shown a very good mitigating ability and ASR expansion seems to be significantly reduced by using the proposed ASR suppressors even with high recycled glass content as shown in Figs. 10 and 11.

It was noticed that expansion of group 1 was extremely higher that allowable limit of the definition of non-expansive aggregate, hence using recycled glass as sand replacement in

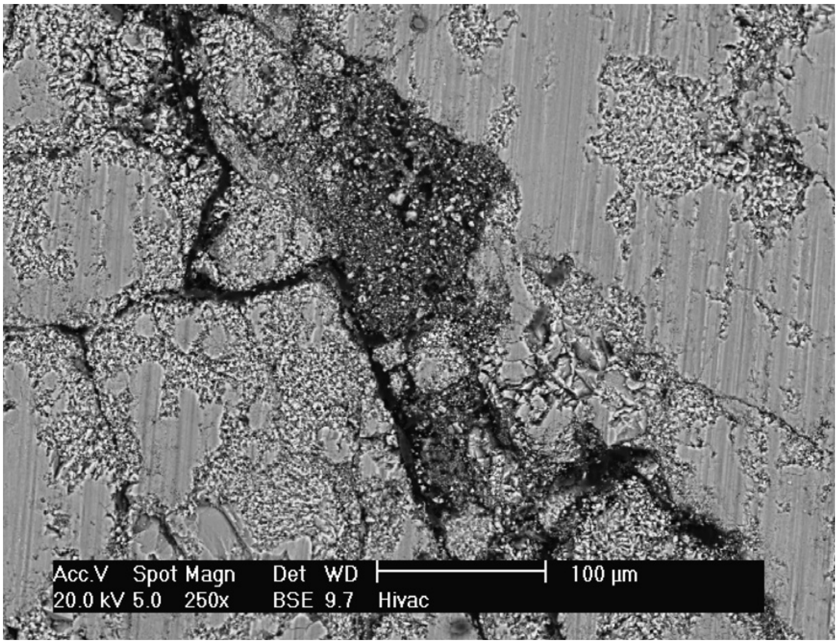
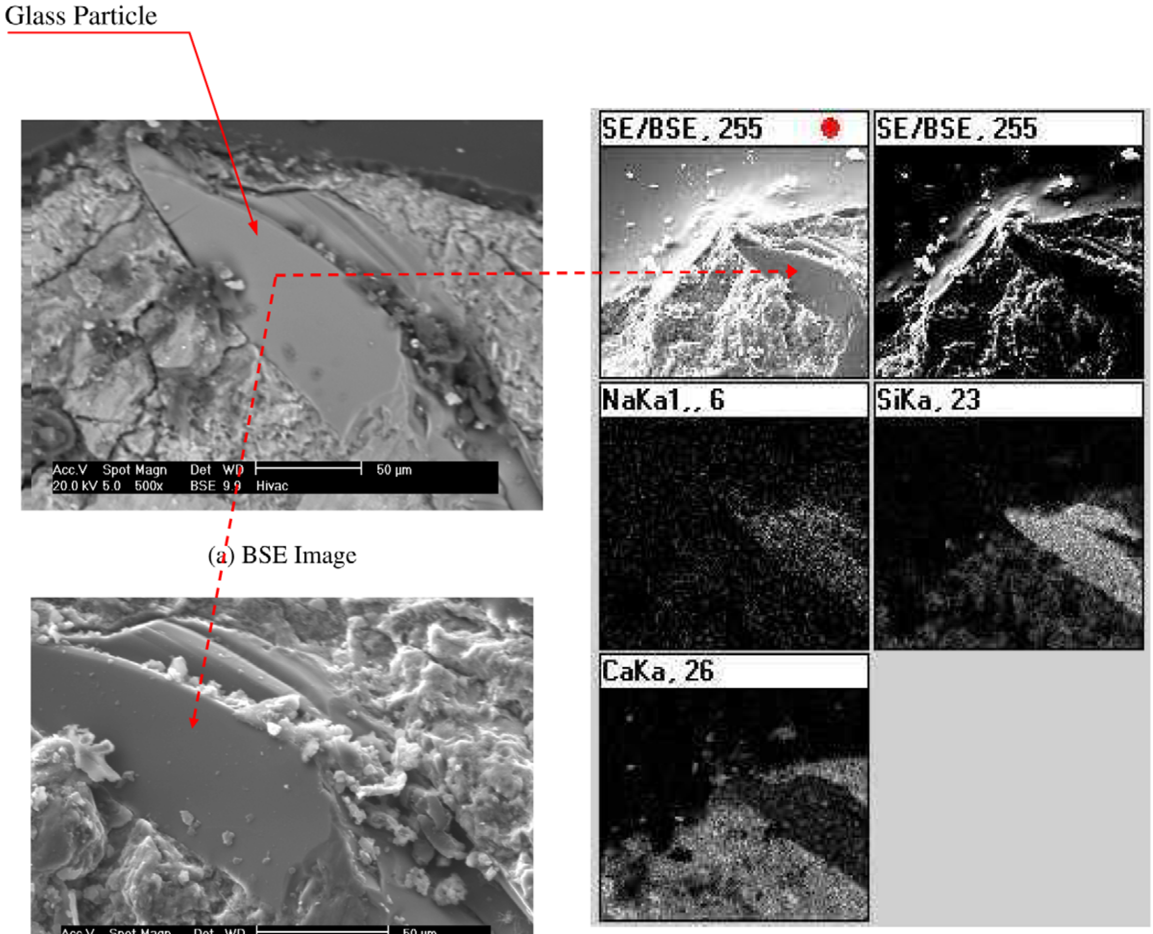


Fig. 12. The formation of ASR cracks in concrete mix M1-2 after 52 weeks.

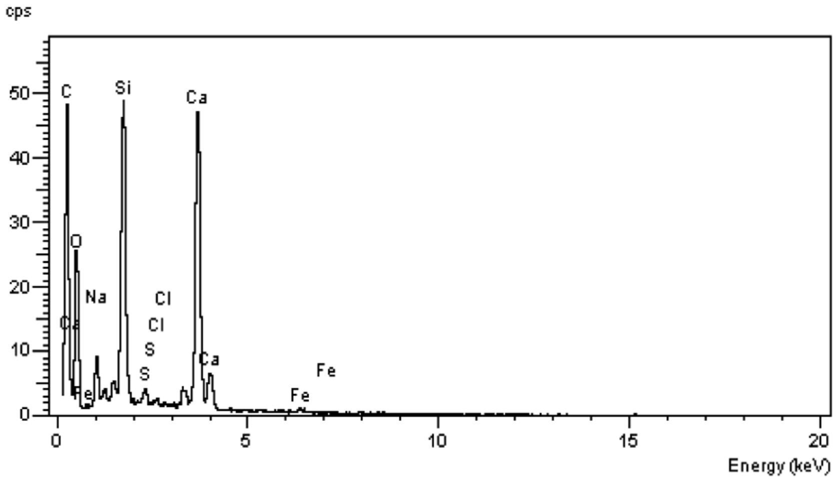
the concrete mix without incorporating any ASR suppressor is not a recommended practice as shown in the images produced by scanning electron microscope Figs. 12 and 13. On the other hand, the expansion monitored was considerably reduced below the maximum allowable expansion limit in concrete mixes of group 2 when 20% of pozzolanic glass powder was used as cement replacement. The explanations of this finding can be concluded in the following:



(a) BSE Image

(b) SE Image

(c) SEM Mapping



(d) EDX-Ray

Fig. 13. Severe ASR damage around glass particle, M1-2.

Table 7
Chemical composition of concrete mixes after one year

Concrete mix ID	Group1 – (Control)			Group 2 – (PGP)			Group 3 – (Li)		
	M1	M1-1	M1-2	M2	M2-1	M2-2	M3	M3-1	M3-2
CaO	42.79	44.67	42.54	32.42	41.78	41.98	43.23	45.15	44.54
SiO_2	17.13	21.4	15.43	18.18	24.85	23.21	17.95	18.84	16.09
MgO	4.8	1.82	5.17	0.98	1.28	1.41	1.75	1.75	1.84
Al_2O_3	1.64	1.84	1.52	1.29	1.66	1.52	1.51	1.55	1.48
SO_3	1.33	1.59	1.33	0.80	1.13	1.07	1.42	1.37	1.47
Fe_2O_3	0.97	1.06	0.69	0.77	0.85	0.62	1.02	0.81	0.83
Na_2O	0.24	1.52	2.38	0.86	2.67	3.91	0.13	2.95	1.40
K_2O	0.28	0.33	0.29	0.25	0.30	0.31	0.27	0.33	0.28
TiO_2	0.06	0.07	0.05	0.05	0.07	0.06	0.06	0.06	0.06
MnO	0.04	0.03	0.03	0.02	0.02	0.02	0.03	0.02	0.02
P_2O_5	0.05	0.05	0.04	0.04	0.04	0.03	0.05	0.04	0.04
LOI	30.24	25.55	29.6	41.95	25.00	25.52	28.11	26.65	28.83
Total	99.57	99.93	99.07	97.61	99.65	99.66	95.53	99.52	96.88
CaO/SiO_2	2.5	2.09	2.76	1.78	1.68	1.81	2.41	2.40	2.77

- Changes in the phase of the chemical reaction.
- Changes in the concentration of the OH^- in the pore solution.
- Lower Ca/Si ratio of the hydration product.
- Lower amount of $Ca(OH)_2$ produced from the hydration of CEM1 and PGP blend.

Fig. 13 shows the ASR expansion damage around recycled glass sand particles occurred in concrete sample extracted from concrete mix M1-2, by using scanning electron microscope (SEM). The SEM mapping of the elements (Na, Si, and Ca) and the energy dispersive X-ray (EDX) for the same sample were also shown in the same figure.

9. Parameters affect ASR

The ASR expansion and the accompanied deterioration expansion can be considered as function of different parameters. From the outcomes of this study, it can be concluded that the formation of an ASR crack is a dependant of different parameters as shown below (Eq. (1)).

$$\mathrm{ASR}_{\mathrm{crack}}: \quad f(\mathrm{A}, \mathrm{R}_{\mathrm{Si}}, \mathrm{OH}^-, m, t, D_{\mathrm{Sol}}, \mathrm{Gel}_{\mathrm{Nat}}, E_{\mathrm{c}}) \tag{1}$$

where A is the alkali content (Na and K), R_{si} is the content of the reactive silica in the aggregate, OH^- is the concentration of the hydroxide ion in the pore solution, m is the moisture, t is the time, D_{Sol} is the degree of solubility of the reactive silica, which depends on the degree of crystallinity and particle size of the parent aggregates, $\mathrm{Gel}_{\mathrm{Nat}}$ is the ASR gel nature (such as thin and easy to permeate or thick), $E_{c:}$ is the modulus of elasticity of the concrete mix that can absorb the internal pressure induced by the ASR expansion.

10. X-ray fluorescent (XRF) and X-ray diffraction (XRD)

The chemical composition of the concrete mixes was monitored after one year by using XRF (Table 7). The XRF figures showed that the lower calcium and slightly higher silica content were found in Group 2. The calcium to silica ratio is shown in Fig. 14.

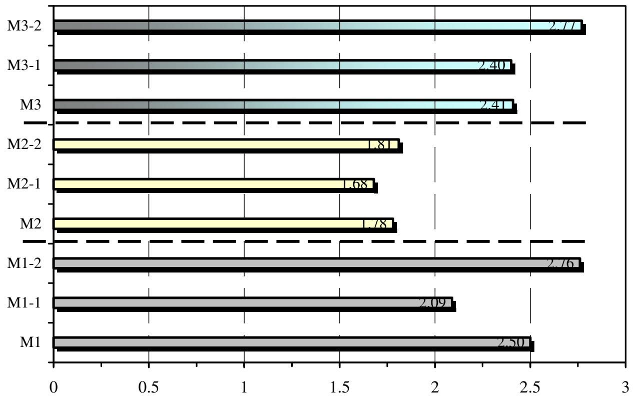


Fig. 14. Calcium to silica ratio CaO/SiO_2 in concrete mixes.

11. Conclusions

1. The mineralogy of recycled glass sand (RGS) is considered as an amorphous crystalline structure which is classified as highly reactive siliceous aggregate when used in concrete.
2. Severe damage can be occurred when RGS is used in concrete mixes without any precautions, such as adding low calcium ashes to replace the normal cement CEM1 or using suitable chemical admixture to minimise ASR, such as lithium compounds.
3. The presence of low calcium content ash in concrete produces ASR gel of high viscosity, thinner than the ASR gel of normal CEM1 concrete. This can make the ASR gel able to permeate and gently distribute into concrete microstructure which reduces the risk of the ASR expansion locally.
4. The presence of pozzolanic glass powder (PGP) in concrete as cement replacement leads to changes in the concentration of hydroxide ion OH^- in the pore solution

which is considered as direct reason in reducing the risk of ASR expansion.

5. The hydration products of low Ca/Si ratio of the low lime ashes can effectively bond the alkalis in the hydration products and C–S–H structure to compensate the lack of calcium in concrete. Therefore, the alkalis will not be free to contribute in the ASR.
6. As the energy required dissolving the PGP is fairly lower than the energy required initiating the break and dissolving the larger glass particles (i.e. RGS). Therefore, the dissolved reactive silica of the PGP and the alkali will contribute to form the C–S–H gel during the early ages of the concrete mix, and both of the dissolved reactive silica and the sodium available in the PGP will be accommodated in the available space of the structure of the hydration products.
7. In spite of the fact that the alkali content in PGP is fairly higher than the CEM1, but the ASR expansion resulted in concrete mixes with the presence of PGP was extremely lower than that of concrete mixes only contain CEM1. The explanation of this phenomenon can be referred to the consumption of the alkali in the structure of the C–S–H gel during the pozzolanic reaction which occurs earlier than ASR, therefore, there will not be enough alkaline to induce ASR.
8. Lithium nitrate is chemically classified as an alkali metal and can contribute to the total alkali content of the concrete. In this study it was approved that lithium nitrate as concrete chemical admixture can successfully reduce ASR expansion.
9. The proposed ASR mitigating mechanisms of the lithium can include the following:
    (a) Different nature for both the ASR gel and the C–S–H gel.
    (b) Reduce the dissolution ability of the reactive silica by applying coating action of the surface of the aggregate particles.
    (c) Reduce the opportunities for the dissolve reactive silica to re-polymerise and form ASR gel.
    (d) Increase the repulsive force between the particles of the ASR gel. Therefore, reduce its expansive ability.

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