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Mitigation Effect of Waste Glass Powders on Alkali–Silica Reaction (ASR) Expansion in Cementitious Composite

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Abstract

The effects of different contents and particle sizes of waste glass powder on alkali–silica reaction (ASR) expansion of cementitious composite bar were investigated in this study. Waste glass powder with particle size less than 300 μm exhibits an excellent mitigation effect on ASR expansion. With larger content and smaller particle size, the mitigation effect of waste glass powder on ASR expansion gradually increases. The mitigation effect of waste glass powder with particle size ranging from 38 to 53 μm and 20% by weight of cement seems relatively better than that of fly ash. When the waste glass powder content reaches 30%, the mitigation effect is still effective and almost the same as that of fly ash. However, the waste glass powder with particle size larger than 300 μm presents negative mitigation effect on ASR expansion when the replacement rate is larger than 30%. On the other hand, the waste glass powder and calcium hydroxide (CH) further react, and produce more calcium–silicate–hydrate gels, which apparently reduce the amount of CH. Moreover, the increasing content of waste glass powder results in a lower pH value in the pore solution of cementitious composite.

Keywords: waste glass powder, cementitious composite, alkali–silica reaction (ASR) expansion, mitigation, pH value

1 Background

Waste glass has considerably contributed to the total solid wastes, which causes the awful waste of resources and serious environmental pollutions (Topçu et al. 2008). It is produced in many forms, including packaging or container glass, flat glass, bulb glass, and cathode ray tube glass, all of which have a limited life in the forms in which they are produced and need to be reused/recycled in order to avoid environmental problems that would be created if they are to be stockpiled or sent to landfills. It is noted that the incorporation of waste glass powder to cementitious composite, mainly cement mortar and concrete, is an effective solution for the recycling and utilization of waste glass (Avila-López et al. 2015; Ali and Al-Tersawy 2012). Theoretically, glass can be a 100%

recyclable material; it can be indefinitely recycled without any loss of quality (Sobolev et al. 2006).

Alkali–silica reaction (ASR) is a deleterious reaction between the alkaline pore solution of concrete and various metastable forms of silica contained in many natural and synthetic aggregates (Rajabipour et al. 2015; Miyagawa et al. 2006). The silica structure is dissolved by the nucleophilic attack of OH^- ions, and the highly degraded silica structure behaves as a hygroscopic silica gel. Alternatively, the dissolved silica can cross-link, coagulate, and form ASR gel. Swelling of this gel leads to stress development and potential cracking of concrete. ASR expansion is a major durability problem of cementitious materials, and continues to damage important infrastructures, including concrete dams and hydraulic structures, pavements, bridges, walls, and power plant structures (Yıldırım and Sümer 2014; Ismail and Al-Hashmi 2009).

Previous studies (Rashad 2014, 2015; Lam et al. 2007) showed that crushed glass used as an aggregate is susceptible to ASR expansion in concrete, because the amorphous silica in glass is dissolved under alkali attack to

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form ASR gel. In contrast, finely ground glass powder as a supplementary cementitious material would prevent ASR expansion, and eventually exhibit certain mitigation effect on ASR expansion (Maraghechi et al. 2014; Shayan and Xu 2006; Shi et al. 2005). This is due to the fact that the glass particle size is crucial for ASR, as the glass powder does not cause ASR issues and can be considered as a pozzolanic material (You et al. 2016; Kim et al. 2014, 2015), while larger particles are potentially reactive in concrete. On the other hand, it is reported that when the particle size of waste glass powder is less than 75 μm , it is unlikely to cause ASR (Corinaldesi et al. 2005). When the particle size of the glass cullet is between 1.18 and 2.36 mm, the ASR expansion of cementitious composite even greatly increases (Du and Tan 2014). Moreover, several studies (Ke and Wang 2014; Shayan and Xu 2004) found that waste glass particles with sizes below 0.30 mm seem not to cause deleterious ASR expansion; whereas waste glass particles with sizes larger than 0.60 mm may lead to the significantly deleterious ASR expansion. However, there are still some arguments on the critical particle size below which the pozzolanic effect of glass powder can outweigh the side effect of ASR damage if the particle sizes are fine enough (Corinaldesi et al. 2005; Shi et al. 2005; Du and Tan 2014; Bažant et al. 2000).

The objective of this study is to investigate the effects of particle size and content of waste glass powder on the ASR expansion of cementitious composite, with the aim to reveal the critical and the optimal particle size, as well as optimal content of waste glass powder. The cementitious composite bar length method was applied to quantitatively assess the mitigation effect of waste glass powder on ASR expansion. Meanwhile, the microstructure characterization and pH value measurement were also conducted to explain the mechanism of mitigation effect on ASR expansion of cementitious composite.

2 Experimental Program

2.1 Raw Materials

Ordinary Portland cement (P.O42.5, from Shaoshan Cement Plant, China) used for specimen preparation had density of 3.06 g/cm^3 , and specific surface area of 296 m^2/kg , which complied with Chinese standard GB175-2007 and was nearly equivalent to ASTM C150 (Standard specification for Portland cement) Type I cement (Xiao et al. 2016). Artificial sands complying with ASTM C1567-13 (2013) (Standard test method for determining the potential alkali-silica reactivity of combinations of cementitious materials and aggregate) were used as reactive fine aggregates, containing mixed quartz and chert were obtained from Xiangjiang River, Hunan, P. R. China. The fine aggregate gradation is given in Table 1. The chemical reagent was 1.0 mol/L

Table 1 Size distribution of artificial sand (fine aggregate).

| Particle size | 300–150 (μm) | 600–300 (μm) | 1.18–600 (μm) | 2.36–1.18 (mm) | 4.75–2.36 (mm) |
|-----------------|---------------------------|---------------------------|----------------------------|----------------|----------------|
| Percent-age (%) | 15 | 25 | 25 | 25 | 10 |

sodium hydroxide (NaOH) solution. The Na_2O powder for NaOH solution preparation was purchased from Yingde Huanbao, Co., Ltd, China. The waste glass powder was made by crushing and grinding from white waste glass bottles by a ball mill for 35 min in the Civil Engineering laboratory at University of South China, China. Then the particles were screened to six size fractions by a sieve shaker including size ranges of 38–53 μm (WG-a), 53–75 μm (WG-b), 75–150 μm (WG-c), 150–300 μm (WG-d), 300–600 μm (WG-e) and 600–900 μm (WG-f). The size distribution of waste glass powder (after 50 min milling) is shown in Table 2. Fly ash (FA) with density of 2.24 g/cm^3 , specific surface area of 376 m^2/kg , and bulk density of 890 kg/m^3 was purchased from Jiahua Power Station, China. The fly ash was classified as Class F by ASTM C618 (2008) (Standard specification for coal fly ash and raw or calcined natural pozzolan for use in concrete) or Grade II by Chinese standards (GBT1596-2005). The chemical compositions of the waste glass powder and fly ash were tested by using X-ray fluorescence (XRF), and the results are shown in Table 3.

2.2 Specimen Preparation

The potential ASR expansion of the prepared cement mortar bars (wet mix method) was assessed in accordance with ASTM C1260 (2014) Standard test method for potential alkali reactivity of aggregates (Mortar-bar method). A total of 21 series of cement mortar bars were prepared. The mix proportion of cement mortar bars for the ASR expansion experiment is shown in Table 4. The mix proportion of control cement mortar bar includes cement (440 g), artificial sand as fine aggregate (990 g) and water (207 g). Portland cement was partially replaced by waste glass powder at percentages of 10, 20 and 30% by weight, and six particle sizes of waste glass powder were adopted for each replacement ratio. Specimens with fly ash substitution at the three aforementioned replacement ratios were also made for comparison. In the experiment, the cement mortar bars were separated into 22 groups labeled by control, WG and FA. WG represents cement mortar bars with six types of particle sizes of waste glass powder. WG-a-10 indicates the cement mortar bar containing waste glass powder with particle size of 38–53 μm and the content is 10% by weight of

Table 2 Size distribution of waste glass powder (after 50 min milling).

| Particle size | <20 (μm) | 20–38 (μm) | 38–53 (μm) | 53–75 (μm) | 75–150 (μm) | 150–300 (μm) | 300–600 (μm) | 600–900 (μm) | >900 (μm) |
|----------------|----------|------------|------------|------------|-------------|--------------|--------------|--------------|-----------|
| Percentage (%) | 11.0 | 18.5 | 27.7 | 22.6 | 9.1 | 8.1 | 1.1 | 0.8 | 1.1 |

Table 3 Chemical composition of waste glass powder and fly ash (% by mass).

| Items | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | Na ₂ O | K ₂ O | SO ₃ | TiO ₂ | Cr ₂ O ₃ |
|--------------------|------------------|--------------------------------|--------------------------------|------|------|-------------------|------------------|-----------------|------------------|--------------------------------|
| Waste glass powder | 70.40 | 3.37 | 0.508 | 9.20 | 1.00 | 13.78 | 0.663 | 0.38 | 0.053 | 0.094 |
| Fly ash | 58.46 | 28.90 | 3.15 | 2.63 | 1.08 | 0.72 | 1.72 | 1.35 | – | – |

cement. Specimen WG-c-30 represents the mortar bar containing waste glass powder with particle size ranging from 75 to 150 μm and the content is 30% by weight of cement. FA represents cement mortar bars containing fly ash replacing cement by 0, 20, and 30% by weight of cement, respectively, for example, FA10, FA20 and FA30. The waste glass powder cement mortar is shown in Fig. 1.

2.3 Test Methods

The experiments were carried out according to ASTM C1567 (Standard test method for determining the potential alkali–silica reactivity of combinations of cementitious materials and aggregate) cement mortar bar length method, and the dimension of each cement mortar bar was 25 mm × 25 mm × 285 mm. After being cast for 1 day, all cement mortar bar bars were demolded and cured in a curing tub with distilled water at constant temperature of 80 °C for 24 h. Then the initial length (L_0) of the specimens was measured using DEMEC-type strain gauges and embedded reference points before they were immersed into NaOH solution for curing. Consequently, cement mortar were immersed in the curing tub with 1.0 mol/L NaOH solution at constant temperature 80 °C, and the length (L_t) was measured at regular intervals for 3, 7, 14, 28 and 35 days, respectively. Afterwards, the ASR expansion of cement mortar bars is calculated by Eq. (1). In this experiment, three duplicated cement mortar bars are used to obtain the average ASR expansion.

$$P_t = [(L_t - L_0)/L_0] \times 100\% \quad (1)$$

where P_t (%) is the ASR expansion of cement mortar bars at the age of T (day); the L_t (mm) is the length of the mortar bars excluding both ends of the length of copper head at the curing age of T (day); the L_0 (mm) is the initial length of cement mortar bars excluding both ends of the length of copper head.

Microstructure characterization of cementitious composite was carried out using a scanning electron microscope (SEM) technique of JEOL 7001F FEG with high

resolution (Li et al. 2017, 2018; Long et al. 2017). This device was equipped with a cold field emission electron gun operating at 15 kV. To examine the internal microstructure of cementitious composite with crashed pieces of hardened specimens obtained after ASR expansion measurement and soaked in acetone to stop hydration. The specimen surface was sputter coated with a thin layer (1.0 nm) of Pt/Pd prior to SEM test. Meanwhile, mineralogical analysis of the hydration products was conducted by XRF. The XRF was used to identify the polycrystalline phases of hardened cementitious composite by means of the recognition of the X-ray patterns that are unique for each of the crystalline phases. The XRF patterns of the cementitious composite show the expected hydration products, including calcium hydroxide and crystallized C–S–H.

In parallel, to investigate the effect of waste glass powder on the ASR expansion of cementitious composite bars, pH values in pore solution of cementitious composite were also measured by solid–liquid extraction method (Cyr et al. 2010). The extraction of the pore solution of small pieces of hardened cement mortar under hydraulic pressure is the most common technique for acquiring pore solution for further analysis including pH measurements (Cyr et al. 2010; Jr and Diamond 1981; Plusquellec et al. 2017). The pore fluid expression device consists of a hollow steel cylinder in which cement mortar specimens are located, a steel piston which is located on the top of the specimen inside the hollow steel cylinder and a steel base plate which has few drainage channels for collecting the extracted cementitious composites pore solution. In terms of pH measurement after extracting the concrete pore solution, pH values using a commercial pH electrode or determining hydroxide ion concentrations can be used for calculating the pH value (Pavlik 2000). Before each extraction test, all surfaces of the device were cleaned by ethanol at least twice until no residue remained. An approximately 6 mm-thick Teflon seal was inserted between the top of the specimen and the bottom

Table 4 Mix proportion design of cement mortar bars for the ASR expansion test.

| Specimens | Portland cement (g) | Waste glass powder (g) | | | | | | | Water (g) | Fly ash (g) | Fine artificial sand (g) |
|----------------|---------------------|------------------------|----------|-----------|------------|------------|------------|-----|-----------|-------------|--------------------------|
| | | 38–53 μm | 53–75 μm | 75–150 μm | 150–300 μm | 300–600 μm | 600–900 μm | | | | |
| Control mortar | 440 | - | - | - | - | - | - | 207 | - | 990 | |
| WG-a-10 | 396 | 44 | - | - | - | - | - | 207 | - | 990 | |
| WG-a-20 | 352 | 88 | - | - | - | - | - | 207 | - | 990 | |
| WG-a-30 | 308 | 132 | - | - | - | - | - | 207 | - | 990 | |
| WG-b-10 | 396 | - | 44 | - | - | - | - | 207 | - | 990 | |
| WG-b-20 | 352 | - | 88 | - | - | - | - | 207 | - | 990 | |
| WG-b-30 | 308 | - | 132 | - | - | - | - | 207 | - | 990 | |
| WG-c-10 | 396 | - | - | 44 | - | - | - | 207 | - | 990 | |
| WG-c-20 | 352 | - | - | 88 | - | - | - | 207 | - | 990 | |
| WG-c-30 | 308 | - | - | 132 | - | - | - | 207 | - | 990 | |
| WG-d-10 | 396 | - | - | - | 44 | - | - | 207 | - | 990 | |
| WG-d-20 | 352 | - | - | - | 88 | - | - | 207 | - | 990 | |
| WG-d-30 | 308 | - | - | - | 132 | - | - | 207 | - | 990 | |
| WG-e-10 | 396 | - | - | - | - | 44 | - | 207 | - | 990 | |
| WG-e-20 | 352 | - | - | - | - | 88 | - | 207 | - | 990 | |
| WG-e-30 | 308 | - | - | - | - | 132 | - | 207 | - | 990 | |
| WG-f-10 | 396 | - | - | - | - | - | 44 | 207 | - | 990 | |
| WG-f-20 | 352 | - | - | - | - | - | 88 | 207 | - | 990 | |
| WG-f-30 | 308 | - | - | - | - | - | 132 | 207 | - | 990 | |
| FA10 | 396 | - | - | - | - | - | - | 207 | 44 | 990 | |
| FA20 | 352 | - | - | - | - | - | - | 207 | 88 | 990 | |
| FA30 | 308 | - | - | - | - | - | - | 207 | 132 | 990 | |



Fig. 1 Cement mortar incorporating waste glass powder for alkali-silica reaction expansion test. **a** Waste glass, **b** specimen after casting.

surface of the piston to reduce the damage to the piston and reduce the friction between cement mortar specimens and piston by facilitating transfer of the applied pressure to the specimens. The loading rate for extraction was about 1–2.8 MPa/s until the maximum 550 MPa stress was achieved. To eliminate residual particles, the collected solution was filtered as soon as possible using filter paper with pore size of 0.45 μm . Then the pH of the pore solution was determined using a precision pH meter. All the liquid specimens were sealed to minimize any carbonation or oxidation.

3 Results and Discussion

3.1 Effect of Particle Size on ASR Development

Figure 2 shows the relationship between particle size of waste glass powder and ASR expansion of cementitious composite bars when the cement was partially replaced using waste glass powder by 10, 20 and 30%, respectively. It can be observed that the ASR expansion of all cementitious composite bars increases as the particle size increases at different curing ages. For instance, all the ASR expansions are less than 0.1% at 7 days curing, and lower than 0.2% at 28 days curing except the specimen WG-f-30, which is just 0.207% when the particle size of waste glass powder is 300–600 μm and the replacement ratio is 30%. At the curing age of 35 days, the ASR expansions of cementitious composite bars WG-e-30, WG-f-20 and WG-f-30 are 0.205, 0.202 and 0.226%, respectively, which are all greater than 0.2%. The result shows that the finer waste glass powder decreases the ASR expansion of the cementitious composite bar, and presents more favorable mitigation effect on the ASR expansion. Coincidentally, the similar trend of ASR expansion was observed when replacing fine aggregate with glass particles in cementitious composite (Cota et al. 2015). If the particle size of waste glass powder is coarser than

300 μm , the waste glass powder seems unlikely to contribute to any mitigation effect on ASR expansion, and even accelerates the ASR expansion when the waste glass powder content is greater than 30%.

3.2 Effect of Waste Glass Powder Content

The relationship between the content and ASR expansion of cementitious composite bars when waste glass powder with different particle sizes is used to partially replace Portland cement is shown in Fig. 3. It can be found that the ASR expansion of cementitious composite bars containing waste glass powder with particle sizes ranging from 38–53, 53–75, 75–150 to 150–300 μm (particle size of 300 μm or less) decreases with the increase in the content of waste glass powder, and all the ASR expansions are lower than the ASR expansion of control cementitious composite bars at different curing ages. The above results illustrate that the higher content of glass powder with particle size less than 300 μm produces lower ASR expansion of cement mortar bars, and has more favorable mitigation effect on the ASR expansion. This finding is consistent with the findings (Shayan and Xu 2006; Taha and Nounu 2008a, b) that fine glass powder can constraint the ASR expansion tendency of reactive aggregates. On the contrary, the ASR expansion of cementitious composite bars containing glass powder with particle size ranges of 300–600 and 600–900 μm (particle size greater than 300 μm) increases with the increase of waste glass powder content, and are all higher than the expansion of the control cementitious composite bars at different curing ages. This indicates that the higher content of waste glass powder with particle size more than 300 μm , leads to higher ASR expansion of cement mortar bars and no any favorable mitigation effect on the ASR expansion.

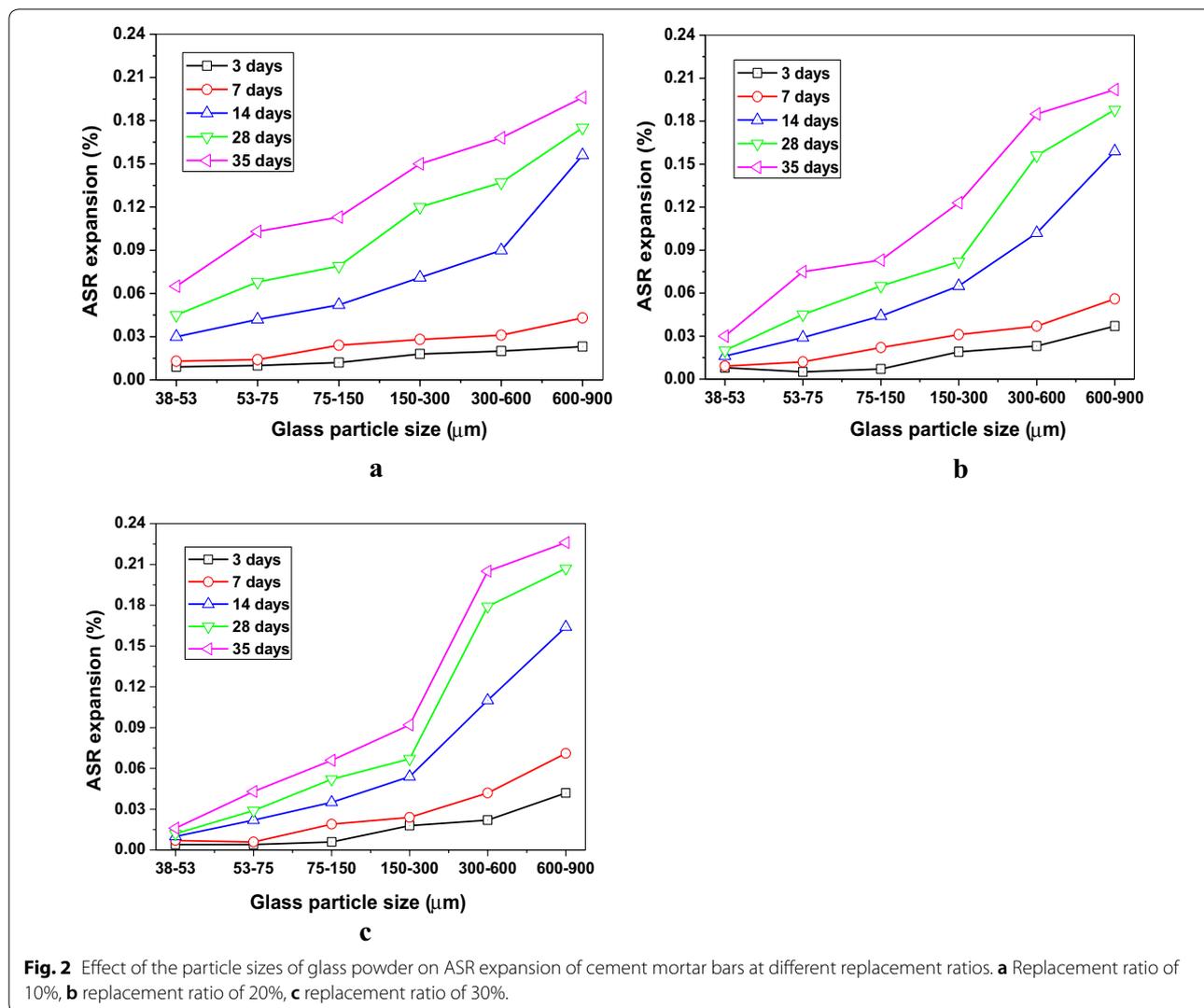


Figure 4 shows the relationship between waste glass powder content and ASR expansion of cementitious composite bars at curing ages of 14, 28 and 35 days, respectively. It displays that the ASR expansions of all the cementitious composite bars containing glass powder with particle size of 300 μm or less are lower than that of the control cementitious composite bars, and decrease with the increase of waste glass powder content. However, the ASR expansions of cementitious composite bars containing particle size greater than 300 μm exhibit the opposite tendency. As shown in Fig. 4a, the ASR expansion of control cement mortar bar is only 0.081% at curing age of 14 days, and the expansions of WG-a-10, WG-a-20 and WG-a-30 are obviously lower than that of the control cementitious composite bar by 62.96, 80.25 and 87.65%, respectively. As shown in Fig. 4b, the ASR expansion of the control cementitious composite bar is

0.126% at 28 days, and the expansion of WG-a-30 is lower by 90.48% than the control cementitious composite bar. However, the ASR expansions of WG-e-30 and WG-f-30 are obviously higher than that of the control cementitious composite bar by 40.06 and 60.29%, respectively. As shown in Fig. 4c, the ASR expansion of the control mortar bars is 0.153% at 35-day curing age, the expansion of WG-a-20 and WG-a-30 is largely lower than the control cementitious composite bars by 80.39 and 89.54% respectively, but the ASR expansions of WG-f-20 and WG-f-30 are significantly higher than the control cementitious composite bars by 32.03 and 47.71% respectively. Clearly, the results indicate that compared waste glass powder with other particle sizes, the waste glass powder with the particle size ranging from 38 to 53 μm presents the lowest ASR expansion at the different curing ages and

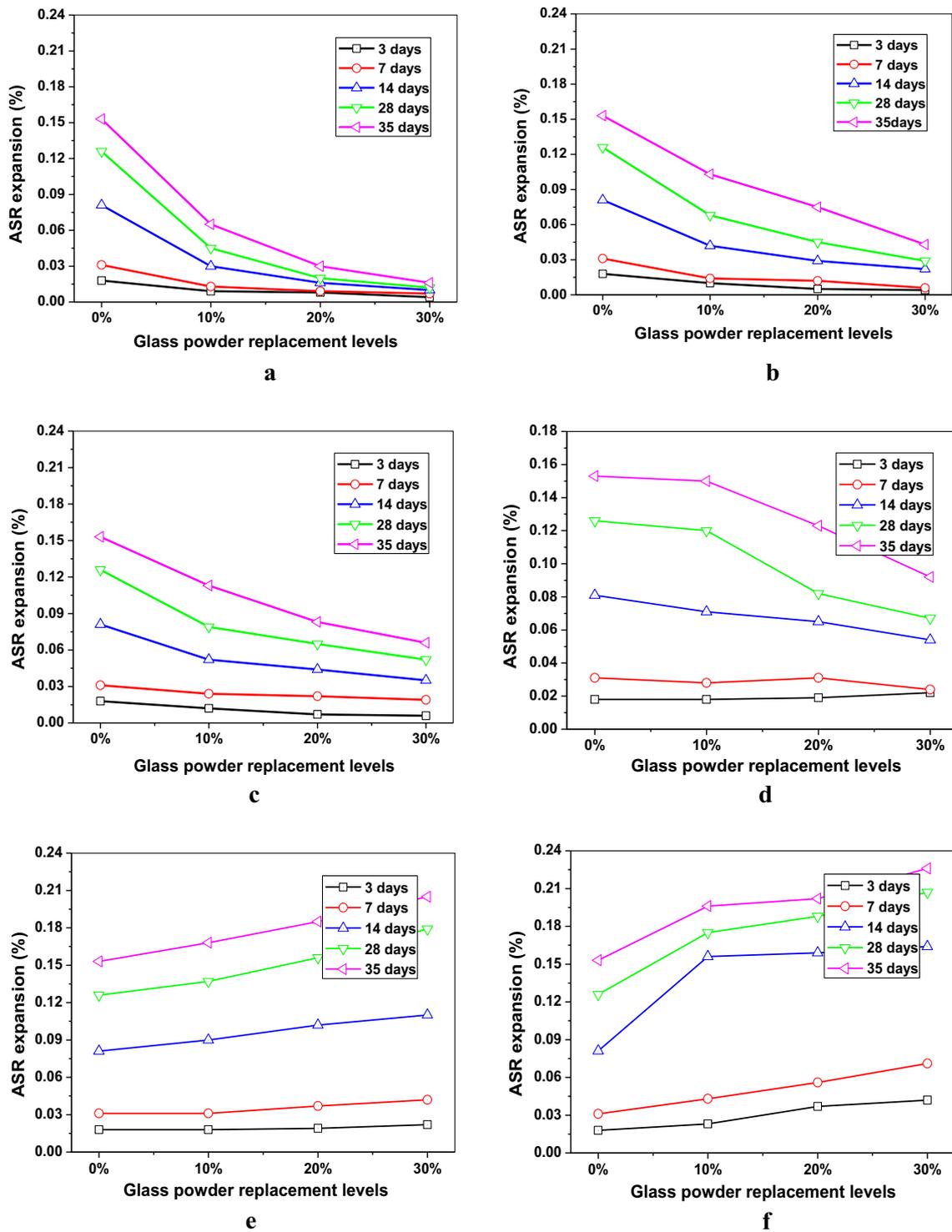
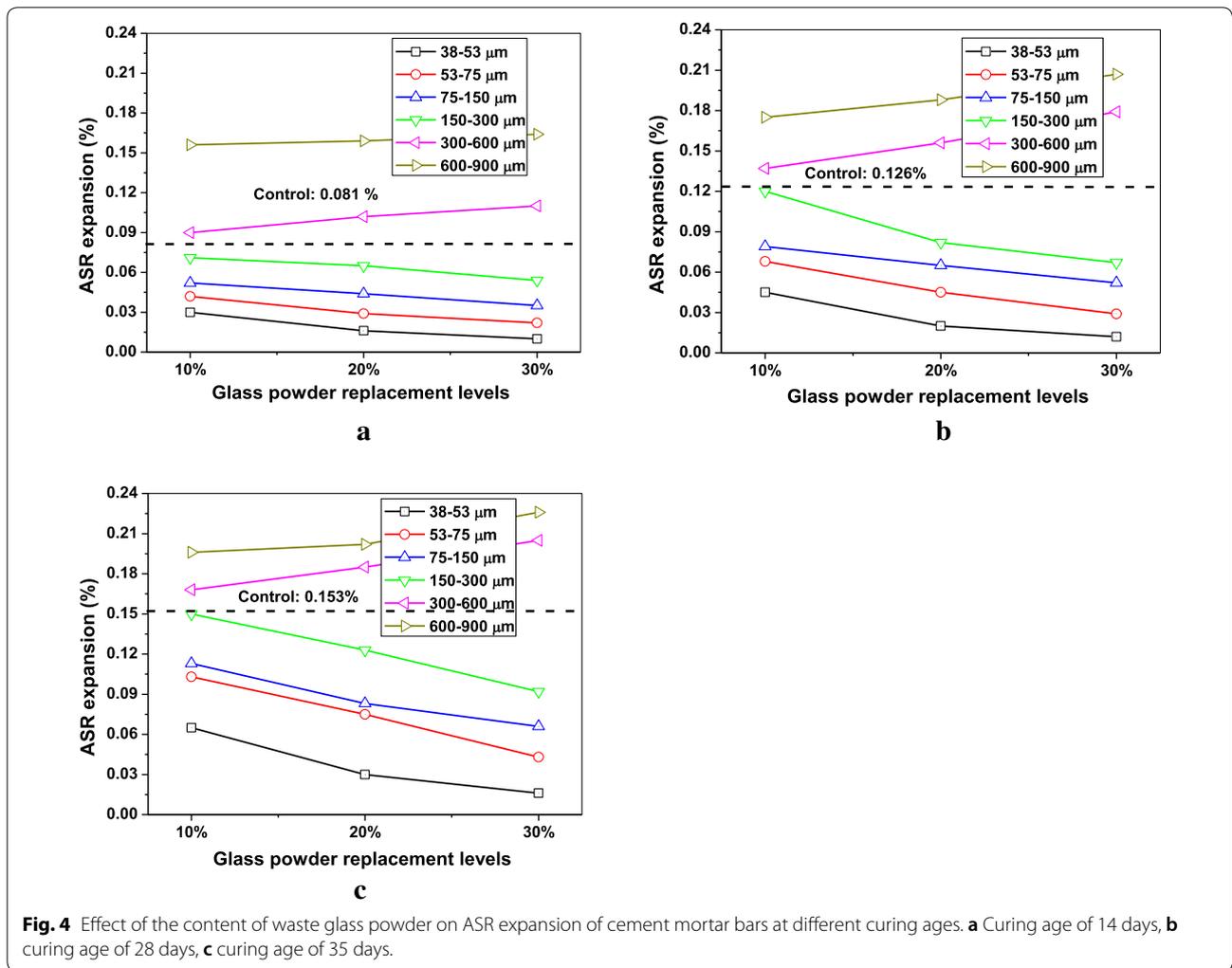


Fig. 3 Effect of the content of glass powder on ASR expansion of cement mortar bars with different particle sizes. **a** Particle size ranging from 38 to 53 μm, **b** particle size ranging from 53 to 75 μm, **c** particle size ranging from 75 to 150 μm, **d** particle size ranging from 150 to 300 μm, **e** particle size ranging from 300 to 600 μm, **f** particle size ranging from 600 to 900 μm.

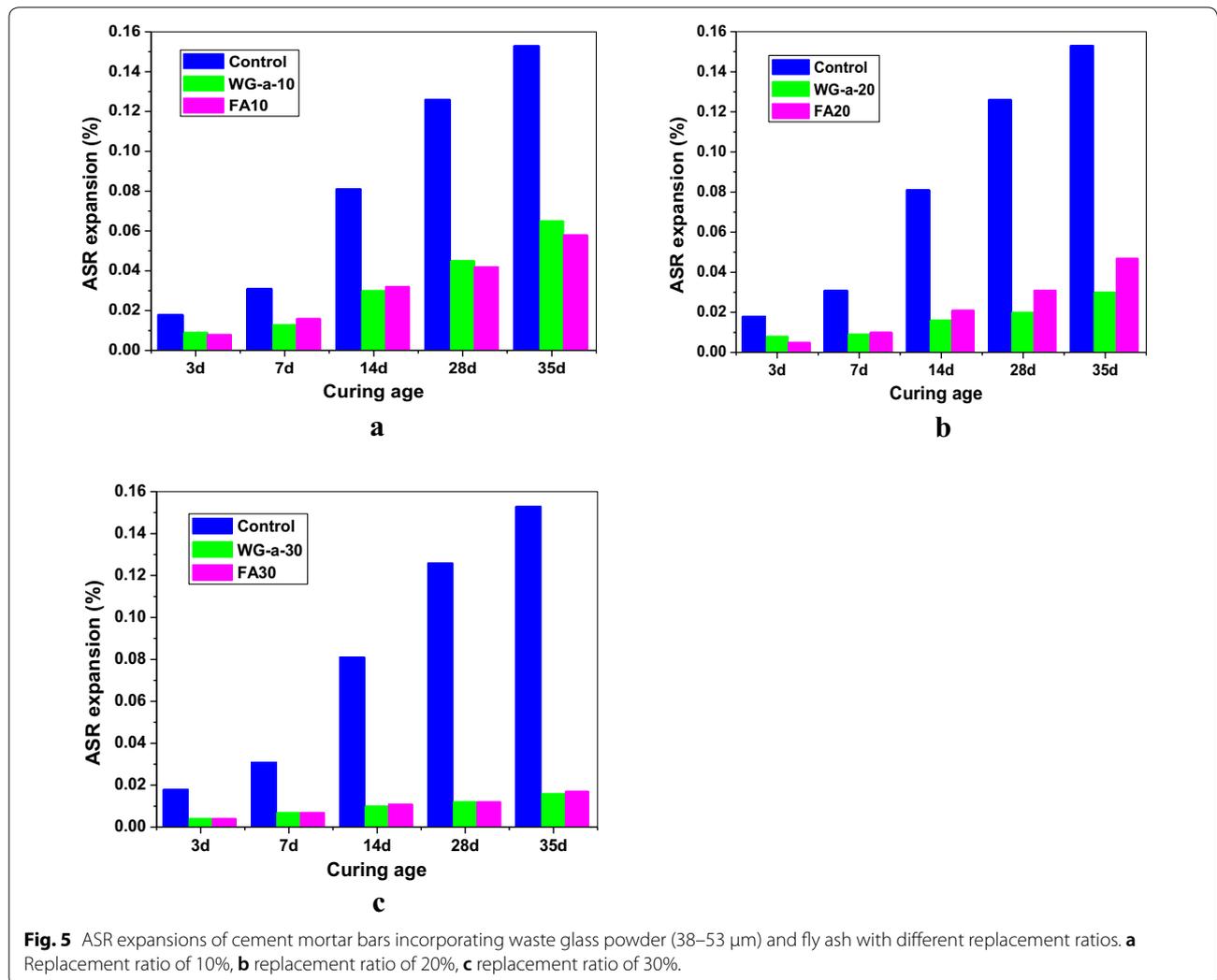


the best effective mitigation effect on ASR expansion of cementitious composite.

3.3 Effects of Waste Glass Powder and Fly Ash

The ASR expansion of cementitious composite bars containing waste glass powder with particle size ranging from 38 to 53 μm or fly ash at different contents is displayed in Fig. 5. It can be seen that the ASR expansion development tendency of cementitious composite bars at different curing ages are similar to each other, and the ASR expansion gradually increases with the curing age, but the ASR expansion of cementitious composite bars containing glass powder or fly ash are obviously lower than that of the control cementitious composite bars. It was found that fly ash is very effective to reduce ASR expansion of cement composites (Shehata and Thomas 2000; Schwarz et al. 2008). For example, from Fig. 5a, with the same replacement level of 10%, the fly ash is more efficient in reducing ASR expansion compared to

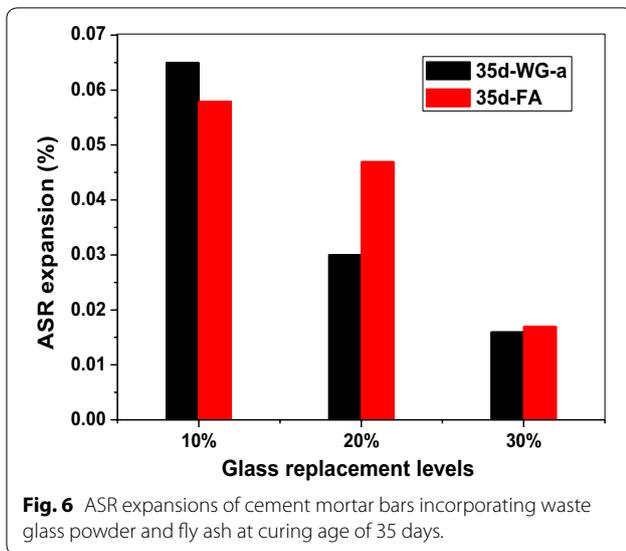
waste glass powder at curing age of 7 and 14 days. However, at the later ages (28 and 35 days), the mitigation effect of waste glass powder on ASR expansion surpasses that of fly ash. As shown in Fig. 5b, at the replacement level of 20%, the ASR expansion of waste glass powder mortar bars is less than that of the specimen incorporating fly ash throughout the curing ages that were tested. At curing age of 35 days, the ASR expansion of the control cementitious composite bars is 0.153%, and the ASR expansion of specimens incorporating waste glass powder and fly ash mortar bars is 0.03 and 0.047%, respectively, which only account for 19.6 and 30.7% of the control cementitious composite bar, respectively. In Fig. 5c, when replacement level is up to 30%, the ASR expansion of the glass powder mortar bars is almost the same to that of specimen incorporating fly ash at different curing ages. For example, at curing age of 35 days, the ASR expansions of waste glass powder and fly ash mortar bars are 0.016 and 0.017%, respectively, which account



for 10.46 and 11.1% of the control cementitious composite bar, respectively. Moreover, waste glass powder with particle size ranging from 38 to 53 μm is superior to fly ash in the mitigation effect on ASR expansion at the replacement ratio of 20%. When the replacement ratio of waste glass powder is up to 30%, the mitigation effect of waste glass powder on ASR expansion of cementitious composite bars is almost the same to that of fly ash. There are more silicates in waste glass, and the amount of chromia (Cr_2O_3) in glass is the most important reason for reduced ASR expansions (Topçu et al. 2008; Du and Tan 2014). The higher waste glass powder content can increase the ASR resistance to some extents, owing to the fact that more waste glass powder and CH reacts to produce more amounts of C–S–H gels because of pozzolanic effect. This can explain the superior mitigation effect of 20% replacement ratio compared to that of 10% replacement ratio. On the other hand, when the content of waste

glass powder increases from 20 to 30%, the beneficial pozzolanic effect is neutralized by the high quantity of amorphous ASR products because of the high content of waste glass powder.

Figure 6 shows the ASR expansion of cementitious composite bars containing waste glass powder with particle size range of 38–53 μm or fly ash at curing age of 35 days. It is observed that the ASR expansion of cementitious composite bars apparently decreases with the increase in the content of waste glass powder, and reaches the lowest value at replacement level of 30%. The ASR expansion of cement mortar bars containing waste glass powder is higher than that incorporating fly ash at replacement ratio of 10%, but it becomes much lower than that of specimen incorporating fly ash at replacement level of 20%. When the replacement level rises up to 30%, the ASR expansions of cementitious composite bars



incorporating waste glass powder and fly ash become merely similar to each other.

4 Microstructure and pH Value Measurement

4.1 Microstructure Characterization

Cementitious composite specimens for microstructure characterization were prepared for the Scanning electron microscopy (SEM) characterization. SEM images of WG-a-10 (waste glass powder particle size range is from 38 to 53 μm and content is 10%) at curing ages of 14, 28 and 120 days are shown in Fig. 7. At curing age of 14 days, Fig. 7a shows that the waste glass powder and calcium hydroxide (CH) react to produce a large amount of C-S-H (calcium-silicate-hydrate) gels. As a result, the amount of CH is obviously reduced. With the increasing curing age, the amount of CH gradually decreases, and the amount of C-S-H gels gradually increases because of the further hydration (Schwarz and Neithalath 2008). As shown in Fig. 7b, c, plenty of C-S-H gels are observed to fill the gaps among waste glass powder gaps and voids, which really contribute to the dense microstructures. The SEM images of FA30 cementitious composite specimen at curing ages of 14, 28 and 120 days are shown in Fig. 8. From Fig. 8a, b, fly ash and reactive CH may have further hydrated to produce a small amount of C-S-H gels in the cementitious composite with the increase of curing age. However, there is still a large amount of unconsumed

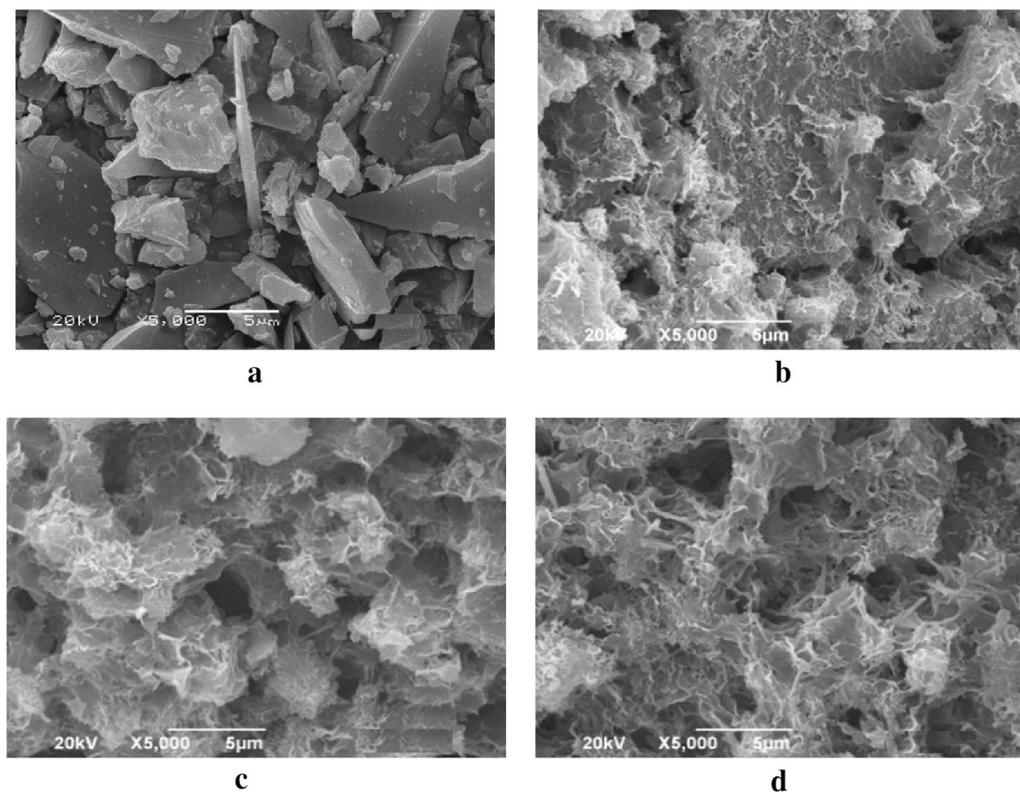


Fig. 7 SEM microstructures of WG-a-30 cement mortar at different curing ages. **a** Waste glass powder, **b** curing age of 14 days, **c** curing age of 28 days, **d** curing age of 120 days.

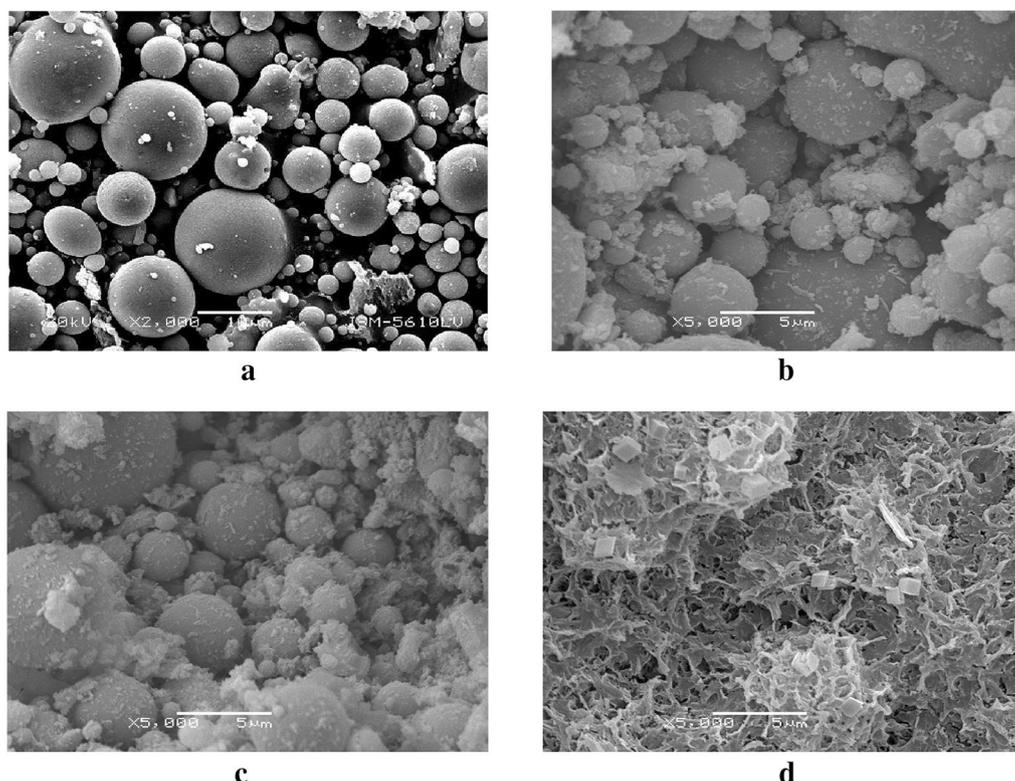


Fig. 8 Microstructure of FA30 cement mortar at different curing ages by SEM. **a** Fly ash, **b** curing age of 14 days, **c** curing age of 28 days, **d** curing age of 28 days.

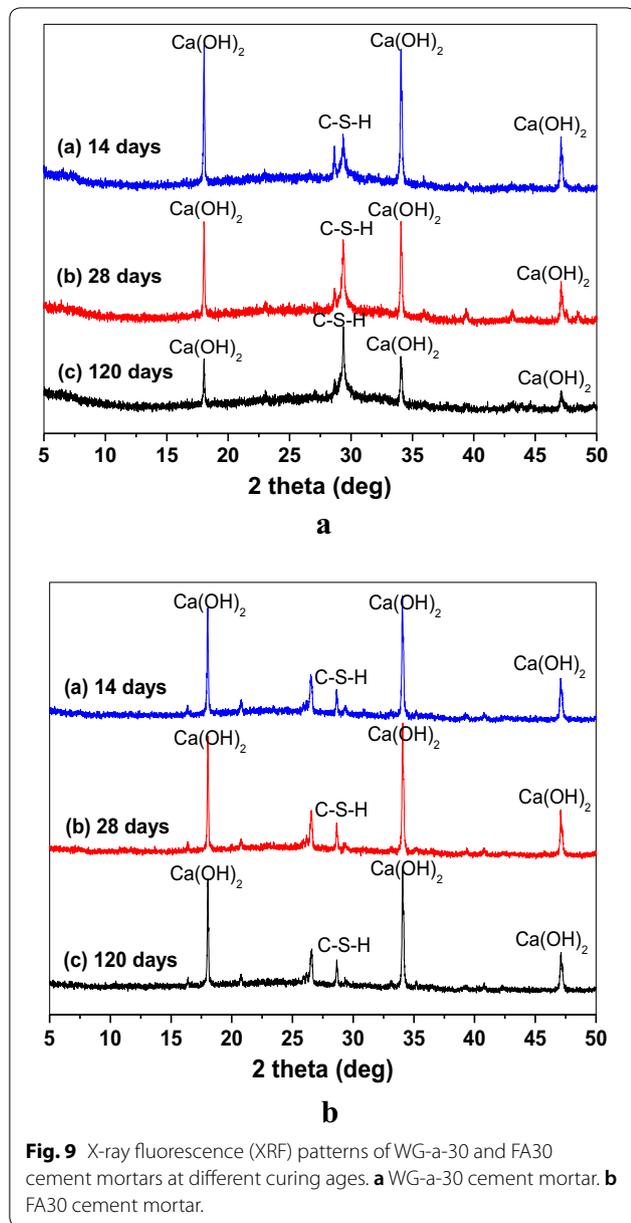
CH crystals are attached to the surface of fly ash particles. In Fig. 8c, it is apparent that some CH crystals attached to the surface of the fly ash particles, and the C–S–H gels fill the gaps between fly ash particles resulting in denser microstructure of cementitious composite. It can be noticed that the microstructures of the WG-a-10 cementitious composite containing the waste glass are remarkably denser than those of the FA30 cementitious composite. Moreover, the WG-a-10 cementitious composite were characterized by a higher amount of hydration products, such as C–S–H gels, with respect to the FA30 cementitious composite, especially at the early curing ages of 14 and 28 days, but at the later curing age of 120 days, this differences became much lower, with a considerable hydration products filling in the pores.

The XRF analysis results of WG-a-30 and FA30 cementitious composite bars at curing ages of 14, 28 and 120 days are shown in Fig. 9. The height of characteristic peak of CH gradually decreases for WG-a-30, while height of characteristic peak of C–S–H gels gradually increases with the curing age. The possible explanations for this phenomenon can be that CH crystals are continuously consumed due to the further hydration reaction with waste glass powder, which produces more C–S–H

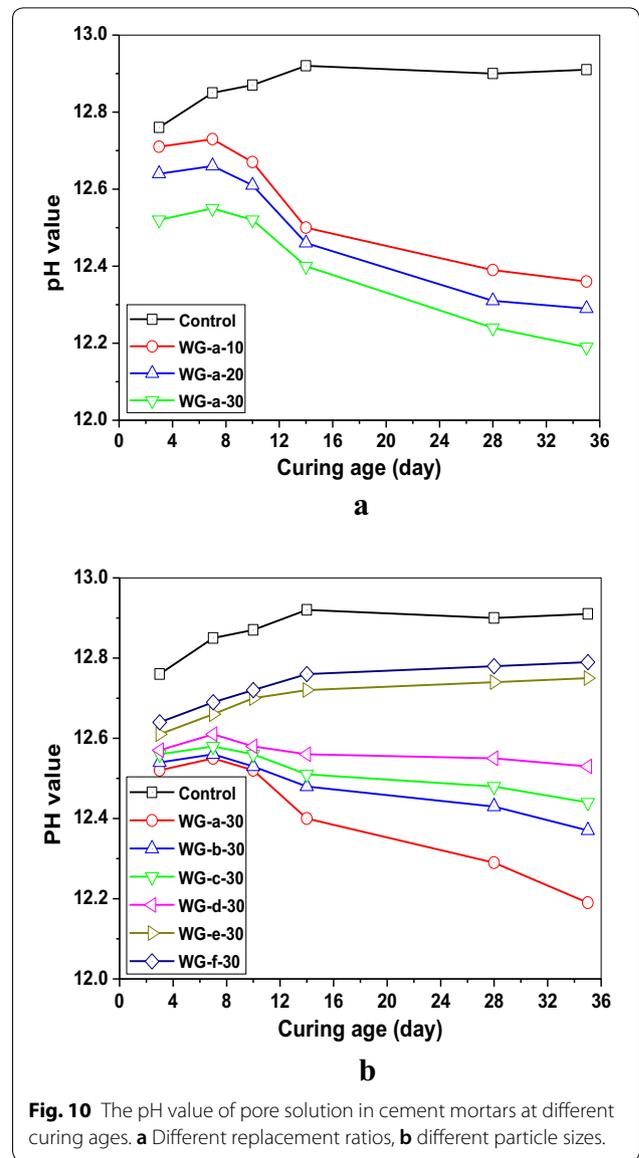
gels. Therefore, the amount of CH in the pore solution decreases, while the C–S–H gels gradually increases, as shown in Fig. 9a. However, for FA30, the height of characteristic peak of CH crystals and height of characteristic peak of C–S–H gels only have minimal changes at different curing ages as shown in Fig. 9b. It indicates that only few CH crystals are consumed by the further hydration between CH crystals and fly ash in FA30.

4.2 pH Value

In order to further investigate the mitigation effect of waste glass powder on the ASR expansion of cementitious composite bars, the pH values in pore solution of control cementitious composite and cementitious composite incorporating waste glass powder with particle sizes ranging 38–53 μm were tested by the solid–liquid extraction method (Nan et al. 2001; Tiruta-Barna et al. 2006). The result shows that all of the pH values concentrate in the range of 12.2–13.1, as shown in Fig. 10. Figure 10a shows the pH value in pore solution of waste glass powder cementitious composite. It is noted that the pH value of control cementitious composite firstly exhibits a gradual increase and then declines to approximate 12.9. However, the pH value of waste glass powder



cementitious composite gradually decreases with the increase of the curing age. It implies that the higher content of waste glass powder results in lower the pH value. Figure 10b shows that the pH value in the pore solution of cementitious composites with different waste glass powder particle size at content level of 30%. The pH values in pore solution of all the cementitious composites are obviously lower than that of control paste, and the pH values of specimens incorporating waste glass powder with particle size not less than 300 μm exhibit a gradual increase with the increase of the curing age, whereas, those of cementitious composites with waste glass powder with particle size not larger than 300 μm show a



opposite tendency. Therefore, the high content of waste glass powder results in lower pH value of the pore solution of cementitious composite. This may be attributed to a higher CH crystals produced by the cement hydration reaction in the control cementitious composite, thus, the pH value was remarkably higher than that of cementitious composite incorporating glass powder at all the curing ages. Prior to the curing age of 28 days, the rapid hydration reaction of alite (C_3S) and belite (C_2S) produce a lot amounts of CH, leading to an increase in pH value in the pore solution of cementitious composite. But the rate of cement hydration reaction tends to slow down after 28 days curing. Therefore, the pH value slowly increases, and gradually stays at a stable level. For cementitious

composites with waste glass powder with particle sizes less than 300 μm , the CH crystals produced from cement hydration reaction are constantly consumed by the further hydration with waste glass powder. Generally, the finer waste glass powder contributes higher pozzolanic activity. As a result, the CH crystals are consumed quickly because of the intense hydration reactions. In addition, the more content of waste glass powder consumes the more CH crystals in cementitious composite. Therefore, at different curing ages, the pH value in the pore solution is lower because the cementitious composite contains more and finer waste glass powder.

5 Conclusions

The mitigation effect on ASR expansion of cementitious composite bars incorporating waste glass powder is investigated in this study. Based on the experimental results and analysis, the following conclusions can be drawn up:

1. When the particle sizes is less than 300 μm , the waste glass powder is obviously effective to reduce ASR expansion of cementitious composite. The smaller particle size and higher content of waste glass powder exhibit better mitigation effect on ASR expansion.
2. However, when the particle size is larger than 300 μm , the waste glass powder seems not to mitigate the ASR expansion of cementitious composite, and even increases the ASR expansion when the waste glass powder replacement ratio reaches not less than 30%.
3. When the content level is 20%, waste glass powder with particle size ranging from 38 to 53 μm is likely to be the optimal for the mitigation effect on ASR expansion of cementitious composite. However, when the content of waste glass powder reaches 30%, its mitigation effect becomes the same to that of fly ash.
4. The amount of CH is reduced because the reaction between waste glass powder and CH produces a large amount of C–S–H gels. Thus, a considerable amount of C–S–H gels is observed to fill the gaps among waste glass powder particles and voids, which obviously contributes to the denser microstructures.
5. CH crystals are continuously consumed due to the further hydration reaction with waste glass powder, which produces more C–S–H gels in cementitious composite. However, only few CH crystals are consumed due to the further hydration between CH crystals and fly ash.
6. The pH value of the control cementitious composite firstly exhibits a gradual increase, and then declines

to around a value of 12.9. However, the pH value of cementitious composite incorporating waste glass powder paste decreases gradually with the increase of curing age, and the higher content of waste glass powder exhibits lower pH value.

Authors' contributions

GK, WL and RL carried out the experiments, YL and GW analysed the test results. WL and YL drafted the manuscript. All authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

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