

## Research Article

# Durability of GFRC Modified by Calcium Sulfoaluminate Cement under Elevated Curing Temperatures

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CSA/GFRC is an advanced composite material possessed with great ductility and durability. However, its bending performance and fibre condition, as well as intrinsic microstructural changes, under elevated temperature have not been understood so far. XRD was applied in this study to investigate the hydration mechanism of CSA cement under 50°C, 70°C, and 80°C. Bending performance was carried out to test the toughness of CSA/GFRC. SEM was applied to observe the underlying microstructural changes of CSA/GFRC under different curing regimes. It was found out that there was a gradual degradation of both ultimate tensile strength and ultimate strain of CSA/GFRC with elevated curing temperature and curing age, but glass fibre still shows considerable ability to carry stress alone by bridging cracks. Microstructural studies showed that, at accelerated temperatures of 50°C and 70°C, the space between fibres remained empty in general only with some hydration products adhering to the fibre surface occasionally. At a higher accelerated curing temperature of 80°C, densification of the interfilamentary spaces by larger and clustered hydration products can be observed at longer curing ages, causing the fibres to lose parts of the flexibility. Therefore, it can be concluded that densification of interfilamentary spaces may have a greater role to play in the strength degradation of CSA/GFRC than mechanisms associated with fibre weakening caused by chemical corrosion.

## 1. Introduction

Glass fibre reinforced concrete (GFRC for short) is a versatile material of considerable potential and has been widely used in engineering over the last four decades [1–3]. However, its long-term durability has aroused some concerns, which has limited its wider application in engineering. It was reported that GFRC suffered from severe strength reduction and ductility reduction as service life increases [4–9]. The exact mechanisms underlying this degradation process are still debated but it is normally accepted that it involves a combination of glass fibre corrosion caused by the hydroxyl in the pore solution [10, 11] and significant CH precipitation between and around fibres that cause loss of flexibility [12].

Calcium sulfoaluminate (CSA) cement is low-carbon cement, possessed with high early strength, good permeability, and improved durability [13–15]. More importantly,

its pore solution is less alkaline than Portland cement, and CH is absent from the hydration products [16]; therefore, it has the potential to produce high-performance GFRC with improved durability properties. Many research studies have been done to investigate the microstructural features and durability of GFRC modified by CSA cement (CSA/GFRC). Due to the limitation of long curing period in the laboratory, most of them are based on hot-water accelerated ageing [4, 17–19]. Purnell and Beddows [18] reported that after curing at 50°C for 140 d, there were almost insignificant changes which occurred in the mechanical performance of CSA/GFRC; with the progress of ageing until 316 d, composite ultimate tensile strength reduced from 30 MPa to 22 MPa, but the embedded fibres could still play an important role in bridging and limiting of cracking development. Research by Song [4] showed that CSA/GFRC retained most of the toughness after ageing for 10 years at

25°C, accompanied by considerably flexible interfacial and interfilamentary areas around the glass fibres.

The previous studies mainly focused on the mechanical property of CSA/GFRC under accelerated ageing. However, the intrinsic hydration mechanism under hot-water curing and its correlation with both microproperties and macroproperties still remain unknown. In the present study, we investigated the hydration process of CSA cement under elevated temperatures. This study aims to clarify the hydration of CSA cement under hot-water accelerated curing and the intrinsic microstructural reasons associated with bending performance changes.

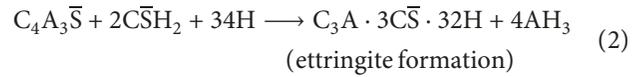
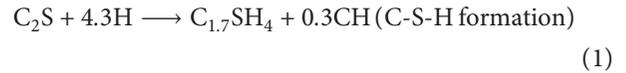
## 2. Materials and Experiment

Chemical composition of the CSA cement used in this study is listed in Table 1. Mixing design of CSA cement mortar is cement: water: sand 1:0.3:1. Cement mortar was cast in plastic tubes and then cured in a water bath set at different temperatures (i.e., 50°C, 70°C, and 80°C). At the investigated curing age, samples were freshly crushed and ground into powder in an agate mortar.

Hydration products of the powder samples were analyzed by XRD. XRD was undertaken by Bruker D8 using CuK $\alpha$ 1 radiation, and the working condition was 40 kV and 30 mA. Data of powder samples was collected in a  $2\theta$ -range from 5° to 80° with a step size of 0.02°, and count duration was set to be 0.5 s per step. Bending performance of CSA/GFRC composites (225 mm  $\times$  55 mm  $\times$  10 mm) was tested by a four-point bending test using Tinius Olsen H25KS, with a major span of 200 mm and a load rate at 1.8 mm/min. Stress-strain curves of CSA/GFRC composites cured at different temperatures were then obtained. SEM was undertaken using JEOL JSM-5800LV, and the secondary electron imaging mode was selected to characterize the morphologies of freshly fractured CSA/GFRC samples.

## 3. Results and Discussion

**3.1. XRD Analysis.** XRD diffraction diagram of CSA mortars cured at different curing regimes is listed in Figure 1. A broad band between  $2\theta=32$  and  $33^\circ$  reveals significant remnants of unhydrated belite clinker even after accelerated ageing at 80°C for 7 d. This may be attributed to rapid water consumption during the hydration of calcium sulfoaluminate within the first 1-2 days, which leaves insufficient water for the hydration of belite at later ages. Hydration of belite is shown in equation (1), generating amorphous C-S-H gels. Ettringite is the main hydration product of CSA cement, generated by the hydration of calcium sulfoaluminate (equation (2)) and is considerably dominant in the CSA sample and conducive to high early strength. It reaches the lowest quantity under 80°C, generating a small quantity of AFm phases. This could be explained that solubility of ettringite increases with elevated temperatures. The XRD diffraction data is also indicative of the presence of quarts in the hydrated CSA mortar.



**3.2. Bending Performance Analysis.** Bending performance of CSA/GFRC cured at different temperatures for 7 d is presented in Figure 2(a). When cured at 50°C for 7 d, CSA/GFRC demonstrated ductile properties with an ultimate strain value of ~0.8%, and its ultimate stress is about 30 MPa. With an elevated curing temperature of 70°C at the same age, there was insignificant changes in the bending performance of CSA/GFRC, and ultimate strain value remains at 0.8% only with a slight decrease of ultimate stress to ~27 MPa. With accelerated curing at 80°C for 7 d, CSA/GFRC composites still exhibited favorable toughness, but glass fibres had lost part of the functions to compensate for the low toughness of the cementitious mortar. According to the stress-strain curve, CSA/GFRC demonstrated 30% strength reduction from 30 MPa to 21 MPa; however, the composite still exhibited great ductility with an ultimate strain of 0.75%.

To investigate long-term bending performance of CSA/GFRC, samples cured at 50°C, 70°C, and 80°C for a longer age of 28 d were also investigated (Figure 2(b)). According to the accelerated ageing model proposed by Purnell, CSA/GFRC samples cured at 50°C for 28 d are equivalent to curing for 438 d under 25°C [18]. Bending performance of CSA/GFRC in Figure 2(b) indicated good flexural toughness. After accelerated ageing for 28 d, glass fibres still played an important role in the bridging effect, and CSA/GFRC exhibited favorable ductility. However, the ultimate strain values of CSA/GFRC demonstrated a medium decrease, and they were 0.746%, 0.789%, and 0.627%, respectively, for CSA/GFRC samples cured at 50°C, 70°C, and 80°C for 28 d, which were 12.0%, 4.6%, and 15.3% lower than those for reference curing regime at 7 d. In contrast, the ultimate strength of CSA/GFRC remained almost unchanged after curing for 28 d compared with 7 d at each curing regime. This is consistent with most of the previous studies [11, 18, 20], which suggested that degradation of GRC composites is a combination of reductions in both strength and ductility as time develops. However, the strength and ductility reduction of CSA/GFRC is considerably gradual and less dominant; therefore, it possesses favorable durability and ductility over ageing.

It is clear that CSA/GFRC indicates advantageous bending performance under elevated curing temperature in the study. It retains most of its toughness, only with a gradual degradation of both ultimate tensile strength and ultimate strain with ageing; however, glass fibre still shows considerable ability to carry stress alone by bridging cracks. This reinforces the confidence in its application in the GFRC industry.

TABLE 1: Chemical composition of the CSA cement (wt.%).

Oxide	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	SrO	LOI
	42.33	9.00	33.82	8.83	1.35	2.29	0.22	0.12	1.61	0.07	0.36

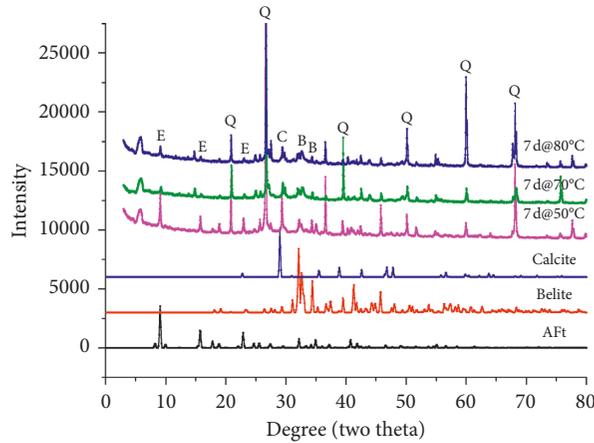


FIGURE 1: XRD diffraction diagram of CSA/GFRC cured at different curing regimes.

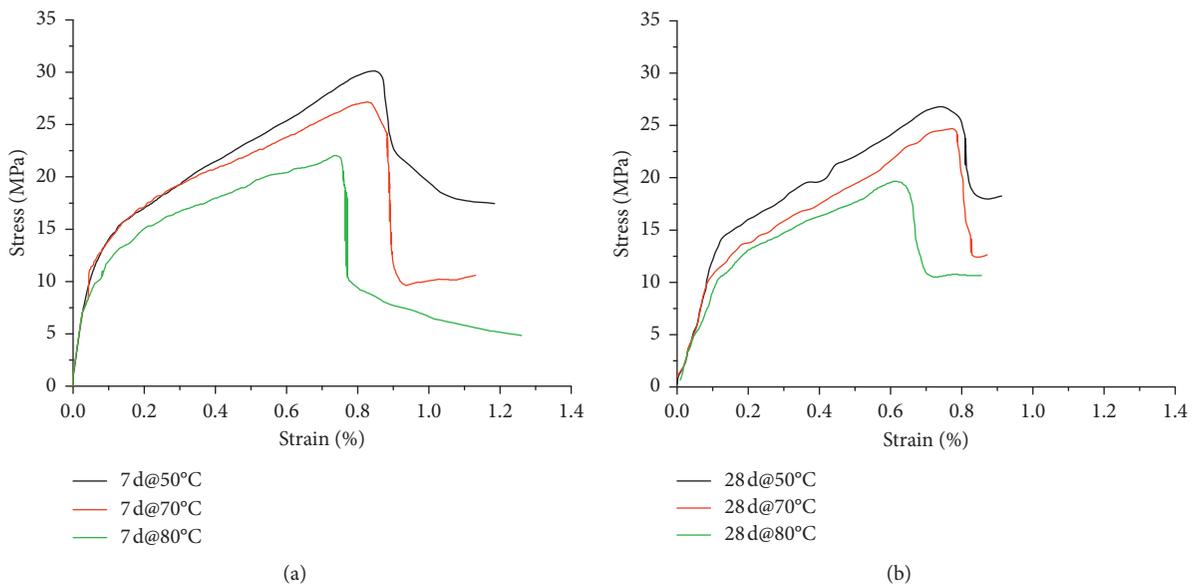


FIGURE 2: Stress-strain curves of CSA/GFRC cured at different curing regimes. Curing day of (a) 7 d and (b) 28 d.

**3.3. Compressive Strength Analysis.** The compressive strength of CSA/GFRC is presented in Figure 3. It indicated a gradual increase in compressive strength with the progress of ageing at each curing temperature. Furthermore, with an elevated curing temperature, the compressive strength of CSA/GFRC demonstrated considerable reduction at each curing age. This can be explained by the solubility of ettringite at elevated curing temperature. As ettringite was the main hydration product of CSA cement and contributed substantially to the strength of the hydrated cement, so solubility of ettringite can lead to the degradation in compressive strength of CSA/GFRC to some extent under elevated temperatures.

**3.4. Microstructure Analysis.** Microstructure on fractured surfaces of CSA/GFRC cured at elevated temperature for 7 d is shown in Figure 4. It can be seen that when cured at 50°C for 7 d, the fibre surface of CSA/GFRC was smooth and intact, only with small amount of needle-shaped hydration products adhering to it, which was supposed to be ettringite generated at early ages. The space between fibres remained empty and was capable to limit propagation of microcracks by bridging effect. With elevated curing temperature at 70°C (Figure 4(b)), there was insignificant changes on the microstructural features of fibre condition; the space between fibres still remained flexural to bridge the crack. However, when CSA/GFRC was cured at 80°C for 7 d (Figure 4(c)),

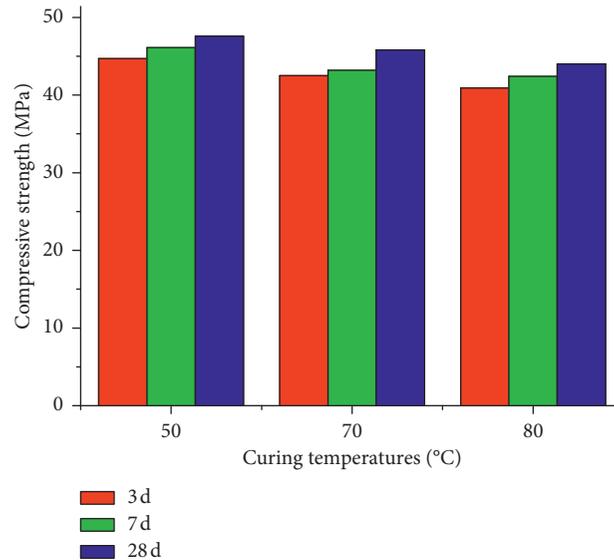


FIGURE 3: Compressive strength of CSA/GFRC cured at different curing regimes.

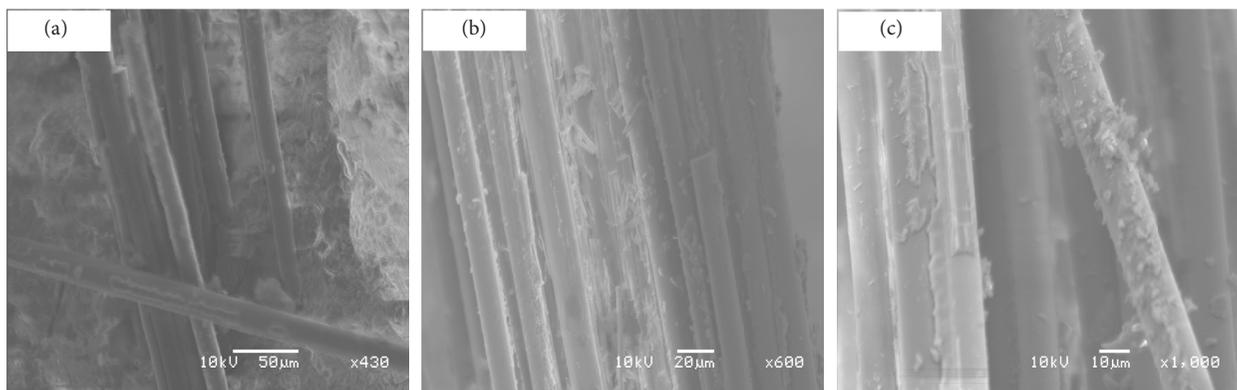


FIGURE 4: Microstructure of the fibre surface in CSA/GFRC cured for 7 d at (a) 50°C, (b) 70°C, and (c) 80°C.

fibre corrosion could not be found and the fibre surface was filled with more densified, larger, and block-shaped hydration products. Meanwhile, the space between fibres still maintained empty in general but occasionally with some products densified between the fibres, such that it led to partial loss of bending toughness in the CSA composite. This is in agreement with the above analysis in bending tests, which suggest that the ultimate tensile strength and ultimate strain values of CSA/GFRC cured at 50°C for 7 d were 30% and 6.2% lower than those for the specimen cured at 80°C for 7 d.

Microstructure on fractured surfaces of CSA/GFRC cured at elevated temperatures for 28 d is shown in Figure 5. It was obvious that when cured at 50°C and 70°C for 28 d, the fibre surface still remained smooth and intact, and severe weight loss was absent on the fibre surface. With an elevated curing temperature of 80°C at the same age, there was no chemical corrosion on the glass fibre. However, the interfilamentary spaces were densified by larger and clustered hydration products. Increased bundle filling may play an adverse effect on the toughness in CSA/GFRC to some

extent, which tended to limit the full potential of ductility compensation of glass fibres [21, 22]. This is in accordance with the bending performance results of CSA/GFRC cured at 80°C for 28 d (Figure 2(b)), which suggest a relative low ultimate strain value of 0.627%, compared with an original value of 0.8% cured at 50°C for 7 d. Moreover, densification of the interfilamentary spaces (especially when cured at 80°C) may lead to enhanced fibre-fibre bond and turn the composite into a more integrated structure. This is the reason why compressive strength reduction of CSA/GFRC is not observed with ageing when cured at 80°C as shown in Figure 3.

To summarize, CSA/GFRC indicates advantageous bending performance under accelerated ageing in the study; glass fibre still shows considerable ability to carry stress alone by bridging cracks. There is only a slight reduction in both ultimate strain and ultimate tensile strength under accelerated ageing at elevated curing temperatures. This reinforces the confidence of the application of CSA/GFRC in engineering where higher ambient temperature exists. Moreover, it would appear that at ageing temperatures up to

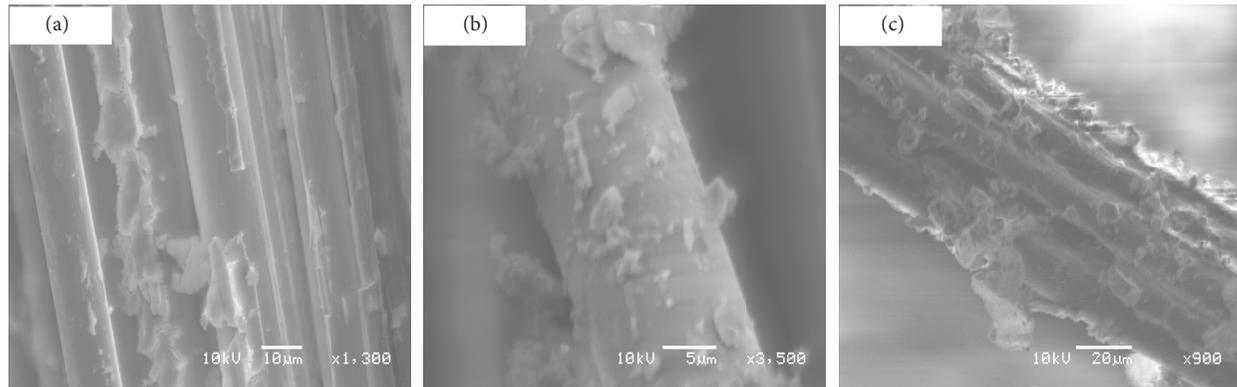


FIGURE 5: Microstructure of the fibre surface in CSA/GFRC cured for 28 d at (a) 50°C, (b) 70°C, and (c) 80°C.

80°C, the key degradation factor of CSA/GFRC performance mechanisms is the densification of interfilamentary spaces (possibly precipitation of ettringite and  $AH_3$  at the space between fibres, etc.) rather than fibre weakening caused by chemical corrosion on the fibre surface.

#### 4. Conclusions

It can be concluded that CSA/GFRC indicates advantageous bending performance under hot-water accelerated ageing, but there is a gradual degradation of ultimate strain value and ultimate tensile strength of CSA/GFRC with elevated curing temperature and curing age. Fibre corrosion is not found in the accelerated ageing process; however, densification of the space between fibres by hydrated products may become a vital microstructural factor in the strength reduction of CSA/GFRC as it causes the partial loss on flexibility. Therefore, studies on how to control the hydration products accumulated on the fibre surface under higher curing temperature is of great importance in controlling the durability of CSA/GFRC composites, and this needs further investigation.

#### Data Availability

The data used to support the findings of this study are included within the article.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

#### Acknowledgments

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