Hardening, microstructure, and shrinkage development of UHPC: A review

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Abstract: Ultra-high performance concrete (UHPC) refers to cement-based materials exhibiting compressive strength higher than 120 MPa, high ductility, and excellent durability. High cracking tendency of UHPC derived from autogenous shrinkage can be a concern due to its high binder content and low water to binder ratio (w/b). This paper reviewed early hardening, microstructure, and shrinkage development of UHPC. The effect of supplementary cementitious materials (SCMs), such as silica fume (SF), ground granulated blast-furnace slag (GGBS), fly ash, limestone powder, rice husk ash, and nano-materials, and curing methods on hydration and microstructure was reviewed. In addition, the effect of cement, water content, aggregate, chemical admixtures, SCMs, curing methods, and fiber on the shrinkage of UHPC was summarized. Different methods to compensate and/or reduce the early-age shrinkage of UHPC were introduced.

Keywords: Ultra-high performance concrete (UHPC); hydration; microstructure; autogenous shrinkage.

1. Introduction

Ultra-high performance concrete (UHPC) refers to cement-based materials with excellent mechanical properties [1-3]. With the incorporation of steel fiber, the ductility and energy absorption of UHPC are typically 300 times greater than that of high performance concrete (HPC) [3]. UHPC possesses excellent durability [4], which is nearly impermeable to carbon dioxide, chlorine ion and sulphates. Its superior durability can increase service life and reduce maintenance [5]. The enhanced abrasion resistance of UHPC provides extended life for structural elements, such as bridge decks and industrial floors. At the same time, it can also provide protection to areas under corrosive or harsh climate conditions [6]. Due to ultra-high compressive strength, UHPC structures weigh only one-third or one-half of conventional concrete structures under the same load [3]. This allows production of more slender structures, which increases usable floor space in high-rise buildings and reduces overall costs. Elimination of steel reinforcement reduces labor costs and provide greater architectural space, allowing nearly limitless structural member shapes and forms for architects and designers [6].

Though UHPC possesses many outstanding properties, the low water-to-binder ratio (w/b) in UHPC and use of high content of fine supplementary cementitious materials (SCMs), such as silica fume, can lead to self-desiccation and even cracking in some cases [7]. Autogenous shrinkage is one of the most important issues that needs to be considered for UHPC, especially at early stage [8]. High autogenous shrinkage was found during the first one or two days after mixing due to the initial hydration, which can cause a considerable cracking potential at early ages. Different research with different mix proportions would result in different shrinkage [9-13]. Autogenous shrinkage up to 1000 με at 28d was reported by Liu [14]. The difference in shrinkage values was related to the water-to-cement ratio (w/c) or water-to-binder ratio (w/b), cement type, types and amounts of SCMs, use of aggregate, curing methods, and fiber characteristics. Therefore, it is very important to clarify the factors influencing autogenous shrinkage of UHPC.

Large shrinkage, especially the autogenous shrinkage, is one of the main characteristics of UHPC, which may induce cracking in the UHPC structures, therefore it is very important to control the shrinkage of UHPC. In addition, the development of shrinkage...
in UHPC is related to the hydration and microstructure of UHPC. Therefore, this paper reviewed hardening, microstructure, and shrinkage development of UHPC in order to provide some insights and suggestions for further research, and to facilitate the applications of UHPC. Factors including cement and water content, aggregate, chemical admixtures, pozzolanic materials, curing conditions, and fiber, were discussed. Some preventive measures for reducing autogenous shrinkage were discussed in order to mitigate shrinkage and improve the cracking resistance of UHPC.

2. Hardening of UHPC

2.1 Hydration process of UHPC

The addition of SCMs has an important influence on hydration of UHPC. UHPC usually contains 10-30% silica fume (SF) based on the mass of total binder materials. It is well known that silica fume could accelerate the hydration of cement in conventional concrete. It also has great influence on the hydration of binder material in UHPC [15] (Fig. 1). As can be seen from Fig. 1(a), compared to the reference mixture U0 (without silica fume, w/c=0.18), the use of silica fume decreases the duration of dormant period of UHPC matrix from 12h to 9h or less. As hydration proceeds, UHPC mixtures with 25% silica fume (U25) shows the accelerated hydration peak first, followed by those of U20, U15, and U10, with 20%, 15%, and 10% silica fume, respectively. However, the hydration peak of U0 is delayed to 28h. The heat of hydration of UHPC mixture with silica fume evolves faster than that of the reference sample [15]. This is due to the fact that the incorporation of silica fume can absorb Ca\(^{2+}\) and OH\(^-\) ions to form calcium silicate hydrate (C-S-H), which increases the rate and amount of heat evolution [16]. However, when the silica fume content increased to 30%, the heat evolution rate decreased and the accelerated hydration period increased [17]. Partial replacement of cement with ground granulated blast-furnace slag (GGBS) or fly ash can retard hydration mainly during the dormant and acceleration periods [17, 18], as shown in Fig. 1(b). However, the combined use of silica fume and fly ash is significantly different from the hydration of pure cement at w/b of 0.35 [19]. This may be due to the reason that silica fume particles dominate the hydration at very low w/b. There is also a slight retardation tendency for the hydration heat of the mixtures with silica fume and GGBS [17]. Limestone powder (LP) can accelerate the hydration of cement [20, 21] and improve the hydration degree of binder materials [22-25]. Use of rice husk ash (RHA) also increases the degree of hydration of cement at later ages because of the pozzolanic reaction and internal curing, even higher than those containing silica fume at 91d [26, 27].

![Fig. 1](image1.png)

**Fig. 1** Heat evolution rate of UHPCs: (a) with different silica fume contents (U0, U10, U15, U20, and U25 represent use of 0%, 10%, 15%, 20%, and 25% silica fume by the total mass of binder materials, respectively) [15]; (b) with different contents of silica fume and fly ash [18].

It is well known that nanomaterials can provide significant enhancement in performance of cement-based material given their physical (filling and nucleation) effects as well as the chemical reactivity. The addition of nanomaterials not only shortened the dormant period but also led to an earlier appearance of the acceleration periods, which have been reported by some references [28, 29]. According to reference [11], the dormant period of the reference mixture is around 13h, the addition of nano-CaCO\(_3\) shortens the dormant period to about 9h due to the filling and chemical effects induced by nano-CaCO\(_3\) [30]. However, the addition of 4.8% nano-CaCO\(_3\), by mass of binder materials, causes an earlier and higher heat of hydration than the addition of 6.4% nano-CaCO\(_3\). This might be due to a dilution effect associated with the addition of nano-CaCO\(_3\) when it is used as a substitution of cement. Nano-SiO\(_2\) can also accelerate the heat of hydration due to its filling effect and nucleation seed for the precipitation of C-S-H.
On the other hand, Norhasri [34] reported that the addition of nano metaclay had a retarding effect on UHPC due to increase of surface area from the nano metaclay.

On the other hand, due to the high dosage polycarboxylate superplasticizer (2%–4%, by mass of binder materials) in UHPC, the early hydration of binder in UHPC is usually retarded. The retardation is dependent on the type and dosage of the superplasticizer. In addition, the increase of curing temperature can accelerate the hydration of cement and promote the secondary hydration between SCMs and Ca(OH)₂ [35], which has a great influence on the hydration products (See the next section).

2.2 Hydration products

The hydration products of binder materials in UHPC is similar to those in ordinary concrete (OC). In UHPC, it contains a larger amount of SCMs. Initially, Portland cement hydrates to form calcium silicate hydrate (C–S–H) and calcium hydroxide (CH). Then, SCMs, such as silica fume, GGBS, and fly ash reacts with CH to form C–S–H [36]. The amount of CH is much lower than that in OC at 28 days as shown in Fig. 2, but the pozzolanic reactions are still incomplete. Different SCMs have different influences on CH content of UHPC. When the silica fume content increases from 0 to 50%, the calcium hydroxide content decreases from 7.4% to 6.25% [17]. Both RHA and SF can strongly reduce CH content, and the effect of SF is greater than that of RHA at later ages [26]. However, the effect of GGBS content has limited effect on the CH content [17]. Shi and Wang [17] found that the combination of silica fume and GGBS had negative synergistic effect on the calcium hydroxide content of UHSC, but no explanation was given.

The content of crystalline phases is considerably higher in OC, whereas less amorphous phases were observed in the UHPC as shown in Fig. 2 [36]. Because of no considerable phase carbonation in UHPC, no calcite is measured even after 28 days in this specimen. The variations of ettringite content development between the first day and the second day indicate some conversion of ettringite to monosulfate phase, and that significant amount of aluminate may enter the X-ray amorphous C–S–H phases [36]. When UHPC contains LP constituents, calcium aluminate monocarbonate is preferably formed, the hydration of C₃S is accelerated, and some carboxylate superplasticizer (2%–4%, by mass of binder materials) in UHPC, the early hydration of binder in UHPC is usually retarded. The retardation is dependent on the type and dosage of the superplasticizer. In addition, the increase of curing temperature can accelerate the hydration of cement and promote the secondary hydration between SCMs and Ca(OH)₂ [35], which has a great influence on the hydration products (See the next section).

The increase of curing temperature can accelerate the hydration of cement and promote the pozzolanic reaction between SCMs and CH [35]. Hydration products of cement after curing at 90°C remain amorphous. On the condition of no external SiO₂ source, the hydration of C₃S and C₅S leads to formation of crystalline α-dicalcium silicatehydrate. On the other hand, tricalcium aluminate (C₃A) and tetracalcium alumino-ferrite (C₄AF) form a hydrogarnet phase. The bonding properties of these two phases are rather unfavorable. If finely ground quartz and/or other SiO₂ sources are used, a pozzolanic reaction takes place between CH and SiO₂, yielding crystalline 1.1 nm tobermorite (C₅S₆H₈) as the main product of reaction at temperatures between about 150°C and 200°C. Xonotlite C–S–H (I), C–S–H (II), and α-C₃SH may also be formed at even higher temperature [40]. The formation of both 1.1 nm tobe-
Rmorite and xonotlite can increase strength of autoclaved products [41]. It is clear from Fig. 3 and Table 1 [42, 43] that there is an important effect of temperature on hydration of UHPC. Hydrates formation ratio increases from 10% to 55% when the curing temperature increases from 90°C to 250°C. It is also significant to notice that Q2 increases faster with temperature higher than Q1 (Fig. 3). This indicates that C-S-H average statistical chain length increases with temperature. This point is also clearly demonstrated by statistical average chain length that corresponds to pentamer at 200°C when it is trimer at 90°C or 200°C. Between 200 and 250°C, another microstructural change is observed with the appearance of a Q3 peak at 250°C (Fig. 3). This peak is attributed to the presence of a crystal hydrate, xonotlite, whose structure implies the presence of silicon-oxygen tetrahedra connected to three neighbor tetrahedra (Q3 species). The H/C (H₂O to CaO) ratio of C–S–H of OC is approximately one, while the H/C of xonotlite is 1/6, and xonotlite forms only in the inner part of concrete specimen [44]. The formation of xonotlite in heat-cured UHPC is due to local, large water vapor pressures. However, lower dynamic equilibrium vapor pressures (3 Pa) can totally suppress the formation of crystalline hydration products, and even no xonotlite or other crystalline hydration products forms at 250°C [45].

3. Microstructure development

UHPC has a very dense and uniform microstructure according to following fundamental effects: (1) close packing of solid particles; (2) hydration and pozzolanic reactions of binder; (3) improvement of the interfacial transition zone (ITZ) between aggregates and bulk matrix [2]. The internal microstructure of UHPC mainly includes unhydrated cement claved products, and microstructure of ITZ.

![Graph showing percentages of Q0 to Q4 for specimens with heat treatment at 90, 200, and 250°C for 8h [43].](image)

**NOTE**: Q represents a SiO₄⁴⁻ unit and the degree of connectivity, n is related to the oxygen bonds number between the SiO₄⁻ units (Q4FS for silica fume and Q4CQ for crushed quartz).

<table>
<thead>
<tr>
<th>Sample</th>
<th>H</th>
<th>PSF</th>
<th>PCQ</th>
<th>C</th>
<th>Statistical length</th>
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<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>-</td>
<td>-</td>
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<tr>
<td>C2/90°C/8h</td>
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<td>10%</td>
<td>0%</td>
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<td>Trimer or quadrimer</td>
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<tr>
<td>C2/200°C/8h</td>
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<td>65%</td>
<td>20%</td>
<td>1.65</td>
<td>Pentamer or hexamer</td>
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<tr>
<td>C2/250°C/8h</td>
<td>50%</td>
<td>75%</td>
<td>65%</td>
<td>1.73</td>
<td>Hexamer or heptamer</td>
</tr>
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**NOTE**: Hydrates formation ratio H=\(Q1 + Q2 + Q3\); connectivity ratio C=\(Q1 + 2Q2 + 3Q3\)/(Q1 + Q2 + Q3); pozzolanic activity of silica fume PSF=(Q4FS-Q4FS₀)/Q4FS₀; crushed quartz PCQ = (QCQ4 – QCQ4₀)/QCQ4₀.
3.1 Pore Structure

UHPC is designed by close packing density and use of SCMs. Therefore, it has a very low porosity. For UHPC at water to binder ratio (w/b) of 0.20, its capillary pores become discontinuous when only 26% of cement is hydrated, instead of 54% for HPC (w/c = 0.33) [47]. Figure 4 shows the pore radius distribution, measured using high-pressure mercury intrusion porosimetry, of a high-quality class C45/55 normal-strength concrete, a class C90/105 high-strength concrete, and two UHPC mixes (90°C heat treatment, coarse- and fine-grained mixes) [48]. The porosity of UHPC consists mainly of pores with diameter smaller than a threshold value (Fig. 5), and a reduction of cumulative porosity corresponds with a decrease in the threshold value. The pore size of UHPC basically concentrates between 2 and 3 nm, the most probable pore diameter is 2.0 nm, and its total porosity is 2.23%.

The curing method and temperature have great influence on pore structure of UHPC. It is nil in the range of 3.75 to 100 nm for UHPC cured at between 150 and 200°C [44]. When the curing temperature is from 20°C to 65°C, setting pressure had no influence on the cumulative porosity in the range of 3.75 nm to 100 μm. However, setting pressure is able to eliminate the air trapped in sample and to compact the sample. A portion of free water is also eliminated. In that range of temperature, the porosity threshold is not modified. For curing temperatures between 80 and 200°C, lower cumulative porosity, intermediate (3.75 nm to 100 μm) porosity and threshold pore size is obtained for pressed samples. The greater part (B 90°C/SC, Fig. 5) if not the whole porosity (BQ 200°C/P, Fig. 5) corresponds in that case to pore diameters smaller than 3.75 nm. For temperatures of 250°C and above, the porosity increases, as well as the threshold pore size (see BQ200°C/P and BQF400°C/SC in Fig. 5).

Fig. 4 The pore radius distribution of normal-strength concrete, high-strength concrete and two UHPC mixes (90°C heat treatment, coarse- and fine-grained mixes) [48]

Fig. 5 Cumulative porosity versus pore diameter of UHPC [44]

NOTE: B20°C/SC, Basic formulation heated at 20°C, soft cast (setting pressure, 1 atm); B20°C/P, Basic formulation heated at 20°C, pressed (setting pressure, 30 atm); B90°C/SC, Basic formulation heated at 90°C, soft cast; BQ200°C/P, Basic formulation with crushed quartz heated at 200°C, pressed (setting pressure 625 atm); BQF400°C/SC, Basic formulation with crushed quartz and steel fibers heated at 400°C, soft cast
The porosity of UHPC decreases with the increase of silica fume content due to its filling and pozzolanic effects [17]. When silica fume content increases from 0% to 50%, the 3d porosity decreases from 14% to 12.7%, and the 56d porosity decreases from 10.4% to 8.3%. If the silica fume content is higher than 25% and GGBS content is less than 25%, GGBS has little effect on the porosity of UHPC at 3d. However, GGBS at any content would increase the porosity of UHPC at 56d. The combination of silica fume and GGBS can cause a negative synergistic effect on the porosity of UHPC in reference [17], but no explanation was given. The total porosity of the RHA modified is higher than that of the SF modified sample, but lower than that of the control sample because of greater filler effect and pozzolanic reaction [26]. The use of 0.3% carbon nanofiber (CNF) can reduce the total porosity of the UHPC by approximately 35%, indicating that the presence of CNF can refine the microstructure of UHPC [32].

3.2 Morphology of hydration products

UHPC is composed of aggregates and compact matrix phase, including unhydrated particles, quartz sand, and hydration products. A reactive interface is formed between completely unhydrated core and hydration products. The core plays a role in skeleton to matrix, and greatly enhances the microstructure of matrix phase [49]. Scanning electron microscope (SEM) observation indicates that the structure of hardened paste is very dense due to very low w/b, hydration of cement, and the pozzolanic action of SF and GGBS. The main hydration product C–S–H gel is homogeneous, no Ca(OH)₂ and ettringite could be found [20, 50]. The curing method has a great influence on microstructure of UHPC, as shown in Figs. 6 and 7.

Fig. 6 shows the microstructures of UHPC samples under different curing conditions. From Fig 6(b), it can be observed that there is clear unhydrated binder constituents in UHPC, which are adhered to several hexagonal CH crystals before heat curing, thus leading to relatively loose microstructure compared to UHPC after heat curing. Visible microcracks are also found propagating along the boundaries of the CH crystals in Fig. 6(b). However, the microstructures of the UHPC samples after 10h of heat curing become much denser compared to that before heat curing, as shown in Fig. 6(c). At the same time, it is less likely to observe the CH crystals in the sample after heat curing. Similarly, in Fig. 6(d), the even denser microstructure of sample after 48h of heat curing confirms that the chemical hydration degree is further higher. The reason is that during heat curing, the high-density nano-crystalline layers typically forms calcium was distinctly difficult to detect the extreme-value of the pore distribution curve. This indicates that heat curing can effectively accelerate chemical hydration of UHPC and form more hydrated products to fill up the pores and voids of the internal structure, resulting in denser microstructure and small pores [51].
3.3 Microstructure of ITZ

The microstructure of interfacial transitional zone (ITZ) in conventional cement mortars and UHPC from SEM observations are shown in Fig. 7. It can be seen that the ITZ between aggregates and paste matrix has a high porosity and CH content, and is the weakest part in conventional concrete. However, UHPC has a very dense ITZ without obvious pores [20]. Normally, the microstructure of the ITZ is influenced by the “wall effect” in the vicinity of aggregate surfaces. This region is about 50 μm from the grain surface into the cement paste [54]. However, in reference [26], sand with particle sizes ranging from only 100 to 300 μm is used as the main aggregate. This can reduce the “wall effect” and thickness of the ITZ. Moreover, with such a small sand particle size and a very small thickness of the ITZ, the effect of SCMs, such as SF or RHA, in reducing the ITZ thickness could not be significant. Therefore, the thickness of the ITZ of all samples obtained in this study is similar and very small. However, owing to the low w/b and pozzolanic reactions between CH and reactive mineral admixture, which consumes most of the CH crystals and converts them to C–S–H [1, 55], the ITZ in UHPC seems as dense as the matrix. The homogenous structure is important for the excellent performance of UHPC.
The denser microstructure of ITZ in UHPC is closely related to high temperature curing. When UHPC is cured in the hot water at 90°C for 10h, the hydration products become denser, and basically the existence of calcium hydroxide cannot be seen (Fig. 6(c)). Increased curing temperature could accelerate the hydration reaction of cement and pozzolanic activity, and improve the microstructure of ITZ [44]. Autoclave curing can improve degree of hydration, and a homogeneous, dense microstructure consisting of close networked crystal fibers with length up to one micrometer would occur under the autoclave curing condition. Cracks and small pores are filled with crystalline C-S-H, as shown in Fig. 8. The pore volume was lower than the heat treated specimen and the median pore diameter was reduced to 5 nm when autoclave curing was used. The autoclaved specimens exhibits dissolution processes around quartz grains, which produces a better cohesion between fillers and fine crystalline cement paste (Fig. 9) [56, 57]. In addition, the heat curing and autoclave curing can also improve the microstructure around the steel fiber [35].

4. Autogenous shrinkage of UHPC

4.1 Factors affecting autogenous shrinkage of UHPC

4.1.1 Binder and water contents

Shrinkage of concrete is usually caused by loss of water due to evaporation or by chemical change resulted from the hydration of cement. Concrete with higher w/b has higher drying shrinkage but lower autogenous shrinkage. For UHPC, the w/b normally is less than 0.25, and the autogenous shrinkage accounts for up to 80% [14]. In addition, the increase in cement content and the decrease in water amount would cause a greater autogenous shrinkage [59]. A higher rate of hydration results in higher autogenous shrinkage due to the decreased volume of hydration products relative to their constituents and higher water consumption. These could reduce bleeding and increase the concrete temperature [60, 61]. It has a major role on early-age shrinkage through controlling the amount of free water, and the development of the microstructure and pore system, which consequently affects the capillary tension and meniscus development (autogenous shrinkage). The strains of samples with lower w/c are higher than those with higher w/c, as shown in Fig. 10. Moreover, cement containing more C₃A content (cement B) led to higher strains. In addition, the shrinkage increases with w/c, when the UHPC is made by cement B (8.6 % C₃A content by mass). This confirms the importance of the C₃A for the autogenous shrinkage of UHPC [9].

4.1.2 Aggregate

Aggregates can act as an internal restraint to reduce shrinkage. It can also reduce the volume of cement paste, leading to lower chemical shrinkage [60]. For UHPC, Meng et al. found that the autogenous shrinkage is reduced with increase of sand to binder ratio [59]. However, Xie et al. [62] reported that the autogenous shrinkage increased with increase of sand to binder ratio. Different conclusions for these two references may be attributed by the fact that the two research results choose different test zeros time for autogenous shrinkage. In addition, the particle size and clay content of aggregate have a significant influence on shrinkage of UHPC. Ma et al. [47] reported that the autogenous shrinkage could be considerably reduced by incorporating basalt coarse aggregates with size ranging from 2 to 5 mm. In addition, there was no swelling in the UHPC specimens produced with coarse aggregates after initial shrinkage, while swelling was observed in UHPC without coarse aggregates [63]. It was found that the shrinkage strain rate linearly increased with clay content of fine aggregate [64]. Furthermore, light-weight aggregates (LWAs) with high water absorption were found to reduce autogenous shrinkage as they act as internal curing materials [8, 65-67]. The shrinkage-reducing efficiency in UHPC is related to the particle size [66], content [65, 67], water absorption [68] and pretreatment method [65] of LWA. For UHPC, it is recommended that use of 15-25% fine LWA is optimal content [67, 69].

4.1.3 Supplementary cementitious materials (SCMs)

The type, fineness, and percentage of SCMs have very significant effect on autogenous shrinkage of UHPC. Silica fume was found to significantly increase the autogenous shrinkage due to refinement of pore structure [70, 71]. Similar increased autogenous shrinkage was observed for GGBS [72, 73]. A high level of cement replacement by metakaolin (10%-15%) was found to reduce both the autogenous and drying shrinkage at early-ages, as shown in Fig. 11(a) [74]. This reduction may be a result of the dilution effect of reducing the cement content [75, 76]. Staquet et al. [77] found that use of metakaolin to substitute 2/3 SF would cause a reduction of around 50% in autogenous shrinkage of UHPC at 6d. In addition, the curing temperature had some influences on the shrinkage reducing effects of metakaolin. Significant reduction of early age autogenous shrinkage was obtained by replacing silica fume with metakaolin in specimens cured at 20°C [48]. However, for UHPC cured at 42°C, the total shrinkage measured for a mixture containing metakaolin was negligible compared to those with silica fume or fly ash [49]. However, Song et al. [28] suggested that the combination
of 60% SF with 40% MK significantly decreased the pore size distribution of UHPC into the range of about 5–10 nm compared to the UHPC with 0% and 100% MK, and it may cause a high autogenous shrinkage. However, there is no autogenous shrinkage results in this reference. Incorporation of fly ash could decrease the shrinkage of concrete, because the unhydrated binder material acted as aggregate to restrain shrinkage and it also diluted cement content [78, 79]. In contrast, it was reported that very fine fly ash had a similar effect to that of silica fume [70]. RHA markedly decreased the autogenous shrinkage due to its internal curing effects on restraining the drops of internal relative humidity [80-82], as shown in Fig. 11(b).

![Graphs showing autogenous shrinkage vs. age](image1.png)

**Fig. 10** Autogenous shrinkage vs. age: variation of w/c at constant cement content and variation of cement content at constant water content [9].

NOTE: Cement B contained considerably more C3A (A: 2.0 mass %; B: 8.6 mass %); 16.2% silica fume by mass.
4.1.4 Chemical admixtures

Greater early-age shrinkage of cement and mortar was observed with the addition of superplasticizer (SP) as a result of improving cement dispersion, which consequently increased the rate of hydration [60, 61]. However, Eppers [9] showed that the superplasticizer content had no influence on the shrinkage of UHPC at the age of 28d, although it had a significant effect on the early age behavior. Shrinkage reducing admixtures (SRAs) belong to a class of organic chemicals known as surfactants [84-87]. SRA can decrease the surface tension of the capillary pore solution resulting in a reduction of the capillary tension and led to a significant reduction in autogenous shrinkage of UHPC [88]. Furthermore, SRA lowered the evaporation rate in the matrix and delayed the mass loss in the drying conditions [85, 89]; and hence the drying shrinkage was reduced. Expansive agent (EA) is categorized as iron powder, alumina powder, magnesia, calcium sulfoaluminate, and calcium oxide. Suzuki et al. [90] reported that an autogenous shrinkage of more than 700 millionths would be reduced to zero with the use of EA. However, the expansion action of EA increased water demand. A combination of EA and internal curing exhibits high efficacy in mitigating shrinkage [91], because internal curing could provide more water for concrete. Recently, a new type of internal curing materials, superabsorbent polymer (SAP) was used for reducing shrinkage of UHPC [8, 14, 89], as shown in Fig. 12. SAP has water absorption capacity of up to 1000 times of their own mass [92] and its internal curing function is based on the release of water from saturated SAP into the matrix as the relative humidity drops over time. The effectiveness of SAPs is related to its chemical characteristics [93-95], dosage [8, 96], particle size [97, 98], and w/b of the matrix [99-101].

4.1.5 Curing condition

Curing method, duration, and temperature have a significant effect on autogenous shrinkage of UHPC. Normally, an early-age drying environment would cause a higher shrinkage for normal concrete and high-performance concrete. For UHPC, because of its denser microstructure, the shrinkage caused by drying condition only occurs for about 20% of total shrinkage [14]. However, a remarkable rise in drying shrinkage was observed when high-volume of fly ash and GGBS was used for UHPC to replace Portland cement [73]. In addition, shrinkage increases with the increasing curing temperature after casting [73, 103]. This was because of the acceleration of hydration reactions, which results in higher chemical shrinkage and the nonuniform distribution of the hydration products. The former is considered as the main driving force for shrinkage until an internal rigid skeleton is formed [104-107]. It is reported that the shrinkage of UHPC could not be observed obviously until the curing temperature reached to
50°C during heat curing. While the curing temperature was close to 70°C, the value of shrinkage increased rapidly and reached to 450×10⁻⁶ ultimately as shown in Fig. 13 [108]. At the same time, the steam curing also increased the early autogenous shrinkage [109].

In addition, the hooked fiber was more efficient in restraining shrinkage compared to the straight and corrugated fibers, as shown in Fig. 13(b) [110]. The crimped and harex fibers were more effective in restraining autogenous shrinkage compared with melt extract and hooked fibers, owing to their superior anchorage characteristics [112]. Bouziadi found that polypropylene fiber is more effective than steel fiber in reduction of autogenous shrinkage of high performance concrete [111].

4.1.6 Fiber

As an essential component of fiber-reinforced concrete (FRC) for strengthening and toughening including UHPC, steel fiber can restrain shrinkage cracking. This is because high elastic modulus of steel fiber can reduce crack width and delay cracking propagation during the shrinkage developing process, including autogenous shrinkage. Wu and Shi [110] found that steel fiber volume had a significant effect in restraining shrinkage of UHPC, as shown in Fig. 14. It is found that the use of 2% steel fibers is the optimal dosage for reducing the autogenous shrinkage of UHPC. In addition, the evolution of autogenous shrinkage is influenced by the geometric characteristics of steel fiber, such as aspect ratio and fiber shape [110, 111]. With the increase of the aspect ratio of steel fiber, total shrinkage decreased [111].

NOTE: S, C, and H represent straight, corrugated and hooked fibers, respectively.

4.2 Mitigation methods for shrinkage of UHPC

Typical UHPC mixtures are characterized by low w/b, high cement and SCMs content, and the incorporation of admixtures. A characteristic of UHPC’s is its superior mechanical properties in the hardened state. However, at the moment of application, some difficulties arise, mainly because of its sensitivity to early-age cracking, which are associated
with self-desiccation and autogenous shrinkage. Cracking may lead to reduced strength, decreased durability, loss of prestress in prestressed structural elements, and structural integrity. Thus, it’s important to understand and control autogenous shrinkage of UHPC.

The shrinkage, especially autogenous shrinkage, development of UHPC is related to hardening and hydration process. The capillary tension theory in general explicates the autogenous shrinkage using pore structure, relative humidity, self-stress, degree of hydration, and interface structure [71]. Over time, free water in the matrix gradually decreases due to progressive hydration of cement and SCMs, and chemical shrinkage occurs. This shrinkage appears as absolute volume change before initial setting, and creates capillary pores in the UHPC matrix. With ongoing hydration, the internal relative humidity reduces. Consequently, a large number of pores are formed in the hardened cement paste and the saturation of water in the pores declines. With the change in the saturation state of capillary pore from saturated to unsaturated, the inner concave surface of pore is subjected to an internal pressure. In order to make the concave surface in a state of equilibrium, the capillary tension increases by which autogenous shrinkage takes place. The capillary tension theory can well elaborate the accentuated influence of low water-to-cement ratio and SCMs in autogenous shrinkage of UHPC as they remarkably affect the pore structure, relative humidity, and self-stress, degree of hydration, and interface structure. Although some studies have addressed the effects of pore structure and relative humidity in particular on auto-genous shrinkage, some equations and model were established between the shrinkage stress with capillary pore diameter, internal relative humidity, and elastic modulus [8]. However, the roles of self-stress, degree of hydration, and interface structure are mostly discussed through influence of cement, SCMs, aggregates, and etc. and there is need for more research to consider the effects microstructure of matrix and interface. Based on previous discussion, different methods should be recommended to compensate for and/or reduce shrinkage of UHPC. Table 2 summarizes these different methods. Two or more methods are often combined to reduce autogenous shrinkage.

### Table 2 Shrinkage compensating methods

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>Methods</th>
<th>Examples</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control hydration of binder materials</td>
<td>By adding SCMs to hinder early hydration or reduce hydration heat</td>
<td>Fly ash, metakaolin, and RHA, etc.</td>
<td>[82, 83, 113, 114]</td>
</tr>
<tr>
<td></td>
<td>Reducing hydration temperature rise</td>
<td>Crushed ice</td>
<td>[62]</td>
</tr>
<tr>
<td>Reduce capillary tension</td>
<td>Restrained the drop of internal relative humidity (Internal curing)</td>
<td>Light-weight aggregate (LWA)</td>
<td>[65, 67]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Super-absorbent polymer (SAP)</td>
<td>[14, 89, 102, 103, 115, 116, 117]</td>
</tr>
<tr>
<td></td>
<td>Reduce surface tension of pore solution</td>
<td>Shrinkage reducing admixture (SRA)</td>
<td>[89, 111, 112]</td>
</tr>
<tr>
<td>Physical restrain</td>
<td>Increase tensile strength</td>
<td>Steel fiber and synthetic fiber</td>
<td>[110, 118]</td>
</tr>
<tr>
<td></td>
<td>Hydration product with tendency to volume increase</td>
<td>Expansive agent (EA)</td>
<td>[90, 103, 119]</td>
</tr>
</tbody>
</table>

5. Conclusions and remarks

Based on the above literature review and discussions, the following conclusions can be drawn:

1. The hydration of binders in UHPC is similar to that in OC. Silica fume and nanoparticles accelerate, whereas GGBS or fly ash retards the hydration of binder. When UHPC is cured under 90°C, the average C–S–H chain length increases. If the curing temperature is raised to 250°C, C–S–H will be dehydrated to form xonotlite.

2. UHPC has a very low porosity, especially after heat curing. The microstructure of UHPC was greatly related to the addition of SCMs and curing conditions. Addition of silica fume or RHA could decrease the porosity of UHPC due to their filling and pozzolanic effects. However, the total porosity of the RHA modified sample was higher than that of the SF modified sample. GGBS had
little effect on the porosity of UHSC at early stages and increased the porosity of UHSC at later stages.

(3) The incorporation of nanomaterial as partial replacement for cement appears to be effective in improving the properties of UHPC. It can produce better dispersion and interaction of the reinforcement systems and achieve significantly higher mechanical strength and durability.

(4) The curing method has a great influence on microstructure of UHPC. High temperature curing is beneficial to the pozzolanic reactions between CH from the hydration of cement and supplementary binder materials, such as silica fume, which improves the microstructure. The autoclaved curing specimens exhibited dissolution processes around quartz grains, which produced a better cohesion between fillers and fine crystalline cement paste.

(5) UHPC with low water-to-binder ratios had high autogenous shrinkage. Aggregates can act as an internal restraint to reduce shrinkage, and also reduce the volume of cement paste, leading to lower chemical shrinkage. Shrinkage reducing agent (SRA) can decrease the surface tension of the capillary pore solution resulting in a reduction of the capillary tension, which lead to a significant reduction in the shrinkage of UHPC.

(6) Silica fume and GGBS were found to significantly increase the autogenous shrinkage due to refinement of the pore structure. However, incorporation of fly ash could decrease the shrinkage of concrete because the unhydrated binder material acted as aggregate to restrain shrinkage. Shrinkage increases with the increasing curing temperature. Fiber type and fiber shape also affect the shrinkage of UHPC.

(7) Different methods have been developed to compensate for and/or reduce early-age shrinkage of UHPC, such as internal curing, fiber, SRA, and EA. When the content is proper, some SCMs, such as fly ash, metakaolin, and RHA, also cloud reduce the autogenous shrinkage.

Acknowledgements

The authors gratefully acknowledge the financial support from the Ministry of Science and Technology under Project No. 2018YFC0705400 and National Science Foundation of China under project Nos. U1305243 and 51378196.

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A new concrete frost resistance evaluating method considering moisture content increase under outdoor exposure

Dequin Ma*, Osamu Senbu, and Ryoma Kitagaki

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Abstract: Three kinds of concrete specimens were made and cured in water for two weeks. Then, half of the specimens were conducted with the accelerated freeze-thaw test and the critical degree of saturation test, directly. Besides, the other half were set in the actual outdoor environment for one year before conducting the same tests. In the accelerated freeze-thaw test, there exists a critical mass moisture content which almost equals to the mass moisture content of the critical saturation S_c in the critical degree of saturation test. In addition, the cycle where the critical mass moisture content is reached is defined as the critical freeze-thaw cycle N_c. The effect of different outdoor exposure on concrete frost resistance can be evaluated by N_c. The outdoor exposure has a negative effect on concrete frost resistance.

Keywords: Critical mass moisture content, freeze-thaw, outdoor environment, concrete frost resistance.

1 Introduction

Concrete structures are exposed to different mechanical and physical forms of attacks during their service life. Even though many concrete structures provide excellent long-term performance, a portion of these concrete have recently shown deterioration, especially during freeze-thaw [1]. Nowadays, two concrete frost evaluation tests, the accelerated freeze-thaw test and the critical degree of saturation test are widely used throughout the world. However, there are occasions that the evaluation results by the accelerated freeze-thaw test does not match with that by the critical degree of saturation test [2].

Nokken et. al figured out that different from the specific 2-5 hours in the ASTM standard accelerated freeze-thaw test, the length of the freeze-thaw cycles in the actual outdoor exposure was considerably longer and ranged from 11-206 hours. [3] Besides, the longer freezing period has been found to be more severe to concrete by Stark. [4] Ma clarified that the effect of the different lowest temperatures on concrete frost resistance. The lower the lowest temperature is, the faster the concrete specimen is damaged by frost. [5] Therefore, it is of great importance to evaluate the effect of actual outdoor exposure on concrete frost resistance.

In this paper, three kinds of concrete specimens with different water to cement ratios and air contents were made. The accelerated freeze-thaw test and the critical degree of saturation test were conducted, respectively. The mechanism of concrete suffered from freeze-thaw has been unveiled through comparing the mass moisture content change between the two test methods and a new concrete frost resistance evaluation method has been proposed. Furthermore, the effect of the outdoor exposure on concrete frost resistance has also been clarified by the newly proposed method. Results will hopefully cast more light into the major mechanism of concrete frost damage.

2 Experimental Outline

Table 1 shows the experimental plan. Three kinds of concrete specimens were made and then cured with different curing conditions. Then, the specimens were conducted with the accelerated freeze-thaw test and the critical degree of saturation test, respectively.

In this paper, Type I Portland cement was used as the cementitious materials. Fine aggregate was the sand with a fineness modulus of 2.68 and the coarse aggregate was the crushed stones with the nominal maximum dimension of 20 mm. Table 2 shows the mix proportion of the concrete specimens.

All the specimens were made in the laboratory. The prism specimens with a dimension of $75 \times 75 \times$
400 mm and the cylindrical specimens with a size of $\Phi 100 \times 200$ mm were cast. All the specimens were removed from the mold on the second day and then submerged in tap water at 20°C for two weeks. The specimens then experienced three kinds of curing conditions; curing condition I: conduct the freeze-thaw test directly, curing condition II: place the specimens directly on the roof of a 2-floor building for 1 year, curing condition 3: place the specimen on a platform on the same roof for 1 year. Compared with condition DJ, moisture is hard to accumulate in condition DD, and dry effect in the actual environment is more prominent. The condition DJ and D represents the concrete specimens exposed to the different moisture environments.

Fig. 1 shows the actual curing condition II and III. For the specimen denotation (e.g 35%-1%-N, 35%-1%-DJ, 35%-1%-DD), the first two parameters indicate the W/C ratio and the air content while the last parameter N, DJ and DD represent the specimens under various curing condition I, II and III.

Concrete resistance to the freeze-thaw was evaluated by the accelerated freeze-thaw method according to JIS A 1148 A and the critical degree of saturation method based on the RILEM. In the accelerated freeze-thaw test, the $75 \times 75 \times 400$ mm prism specimens experienced cyclic freeze-thaw cycles. The test was paused at suitable time intervals and the specimens were taken out to measure the weight in the air and water, the expansion by length change and the damage by the relative dynamic modulus of elasticity (RDM). The accelerated freeze-thaw test was ceased when RDM fell to 60% or 300 cycles, whichever came first. Then dried the specimen in an oven at 105°C to constant weight and the weight was measured.

The $\Phi 100 \times 200$ mm cylindrical specimens were used for the critical degree of saturation test. The test was divided into two parts. One part is to determine the critical moisture saturation $S_{cr}$ and the other one is for deciding the potential degree of saturation at moist condition $S_{cap}$. In the $S_{cr}$ test, specimens were firstly vacuum saturated. Then the specimens were dried at 50°C to the desired moisture degree and wrapped with preservative film to experience freeze-thaw cycles. The weight and RDM were measured before and after the cycles. $S_{cr}$ is achieved at the point when RDM decreases dramatically.

The degree of saturation $S_{cap}$ was calculated by the single surface water absorption test in an isothermal room with constant temperature 20°C and relative humidity RH 60%. Samples with a dimension of $\Phi 100 \times 30$ mm were cut from the cylindrical specimens and dried at 50°C for three days. Then all the samples were set in a stainless tray filled with water. The samples were taken out at suitable time intervals to measure its weight. The time $T_{pl}$ when $S_{cap}$ reaches to the $S_{cr}$ is defined as a concrete frost resistance evaluation criterion.

![Fig. 1 – Curing condition II and III](image)

<table>
<thead>
<tr>
<th>Concrete type</th>
<th>Curing condition</th>
<th>Test method</th>
</tr>
</thead>
<tbody>
<tr>
<td>35-1</td>
<td>Condition I: conduct the test directly (N)</td>
<td>The accelerated freeze-thaw test</td>
</tr>
<tr>
<td>55-1</td>
<td>Condition II: set on the roof for 1 year (DJ)</td>
<td></td>
</tr>
<tr>
<td>55-4.5</td>
<td>Condition III: set on a platform on the roof for 1 year (DD)</td>
<td>The critical degree of saturation test</td>
</tr>
</tbody>
</table>
until 54 cycles and then it started to de-
freeze. The mass moisture content increased con-
nuously with the water in the capillary frozen into ice causes the
moisture content change of three types of con-
crete specimens in the accelerated freeze-
thaw test almost equals the Mcr, in the
critical degree of saturation test. Hence, it is sug-
gested that concrete exists a critical mass moisture
content under freeze-thaw action and it is not af-
fected by the different freeze-thaw methods. Con-
tcrete exhibits obvious frost damage when the critical
mass moisture content is achieved.

Besides, since the results of the length change are easy to achieve, and the length change curve has fewer variations than the RDM and mass moisture content. In this paper, the nickpoint in the length change is defined as the critical freeze-thaw cycle Nf and its mass moisture content represents the critical mass moisture content Wcr.

The nickpoints of the length, RDM, and mass moisture content change in the W/C55%-4.5% specimens also appear at similar freeze-thaw cycles, and they almost share identical mass moisture content, which is close to the Mcr. However, the W/C55%-1% specimens show completely different tendency during the freeze-thaw cycles. The curves of the length, RDM and mass moisture content change exhibit dramatic change even from the beginning of the freeze-
thaw cycles, and no obvious nick points can be figured out. As shown in Table 3, even the mass moisture content at 0 cycle W0 has already exceeded the Mcr. Therefore, concrete is damaged by freeze-thaw right after the start of the freeze-thaw cycles. The results of three types of specimens have verified the existence of the critical mass moisture content Wcr during the freeze-thaw cycles.

### Table 2 – Concrete mix proportion

<table>
<thead>
<tr>
<th>w/c</th>
<th>Target air content (%)</th>
<th>Unit amount (kg/m³)</th>
<th>Admixture (Cx%)</th>
<th>Actual air content (%)</th>
<th>Slump (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>W</td>
<td>C</td>
<td>S</td>
<td>G</td>
</tr>
<tr>
<td>35</td>
<td>1</td>
<td>165</td>
<td>471</td>
<td>827</td>
<td>975</td>
</tr>
<tr>
<td>55</td>
<td>1</td>
<td>180</td>
<td>329</td>
<td>936</td>
<td>943</td>
</tr>
<tr>
<td>4.5</td>
<td>0.014</td>
<td>180</td>
<td>329</td>
<td>843</td>
<td>943</td>
</tr>
</tbody>
</table>

### 3 Experimental Results

#### 3.1 The mechanism of concrete under F-T cycles

Enlightened by the moisture degree increase in the critical degree of saturation test, the moisture degree of concrete in the accelerated freeze-thaw test should also increase with the conduction of the freeze-thaw cycles. Therefore, RDM in the accelerated freeze-thaw test should also show an obvious decrease once the critical moisture saturation Scr is reached. In this paper, the mass moisture content of the specimen in the two freeze-thaw tests are calculated and compared with each other to verify the mechanism of concrete under freeze-thaw.

The mass moisture content after N cycles (Wn) in the accelerated freeze-thaw test is listed below.

\[
W_n = \frac{m_{n, air} - V_n \times \rho_{105°C}}{V_n \times \rho_{105°C}} \times 100% \tag{1}
\]

where \(W_n\) is the mass moisture content after N cycles. \(V_n\) is the volume of the specimen after N cycles and \(\rho_{105°C}\) is the density of the oven dry specimen.

The critical mass moisture content \(M_{cr}\) in the critical degree of saturation \(S_{cr}\) is calculated as follows:

\[
M_{cr} = \frac{m_{cr} - V \times \rho_{105°C}}{V \times \rho_{105°C}} \times 100% \tag{2}
\]

where \(M_{cr}\) is the mass moisture content at \(S_{cr}\) and the \(m_{cr}\) is the weight of the specimen at \(S_{cr}\).

Fig.2 shows the results of the length, RDM and mass moisture content change of three types of concrete specimens in the accelerated freeze-thaw test. As shown in the figure, for the W/C35%-1% specimens, the RDM almost kept the same before 54 cycles before it dropped significantly afterward. The length curve also showed similar tendency that the nickpoint of the length change occurred at 46 cycles. The mass moisture content change is also exhibited in Fig.2. The mass moisture content increased continuously until 54 cycles and then it started to decrease. It is reckoned that the expanded volume when the water in the capillary frozen into ice causes the microcracks of the matrix. Therefore, more external water intrudes into the specimens through these microcracks and furtherly exacerbates frost damage. When the saturation degree reaches the critical degree of saturation \(S_{cr}\), specimen will show apparent damage and begin to peel off, which has been figured out by the decrease of the mass moisture content.

The mass moisture contents at the nick point of the specimens in the two tests are illustrated in Table 3. Despite some differences in the W/C35%-1% specimens, the mass moisture contents \(W_0\) at the nick points of the length change correspond with that of the RDM change and the mass moisture content change. Furthermore, the \(W_0\) calculated in the accelerated freeze-thaw test almost equals the \(M_{cr}\) in the critical degree of saturation test. Hence, it is suggested that concrete exists a critical mass moisture content under freeze-thaw damage and begin to peel off, which has been figured out by the decrease of the mass moisture content.
The effect of different outdoor exposures DJ and DD on concrete frost deterioration has been evaluated by $N_f$ in this paper. The specimens after outdoor exposure DJ and DD were firstly cleaned and submerged in water for 2 weeks before conducting the accelerated freeze-thaw test, and the length, the weight in the air and water were also measured at suitable time intervals during the freeze-thaw cycles. The results of the effect of one-year outdoor exposure are listed below.

<table>
<thead>
<tr>
<th>Type (No exposure)</th>
<th>The critical degree of saturation test</th>
<th>The accelerated freeze-thaw test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$M_{cr}$ (mass moisture content of $S_{cr}$)</td>
<td>$W_0$ (%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Length</td>
</tr>
<tr>
<td>35-1-N</td>
<td>6.10</td>
<td>5.87 (34 cycles)$^*$</td>
</tr>
<tr>
<td>55-1-N</td>
<td>7.10</td>
<td>9.80 (0 cycle)</td>
</tr>
<tr>
<td>55-4.5-N</td>
<td>7.08</td>
<td>5.86 (34 cycles)$^*$</td>
</tr>
</tbody>
</table>

$^*$The mass moisture content is calculated from the 34 cycles for the 35%-1%-N and 55%-4.5%-N specimens

### 3.2 The effect of outdoor exposure on concrete frost resistance

According to the results above, the critical freeze-thaw cycle $N_f$ can be regarded as the demarcation point of the concrete damage by freeze-thaw. Thus, the effect of different outdoor exposures DJ and DD on concrete frost deterioration has been evaluated by $N_f$ in this paper. The specimens after outdoor exposure DJ and DD were firstly cleaned and submerged in water for 2 weeks before conducting the accelerated freeze-thaw test, and the length, the weight in the air and water were also measured at suitable time intervals during the freeze-thaw cycles. The results of the effect of one-year outdoor exposure are listed below.

Fig. 3 exhibits the length change of the W/C 35%-1%-DJ and DD specimens after one-year outdoor exposure. Compared with the nickpoint appearing at 42 cycles in the W/C35%-1%-N specimens, the length of 35%-1%-DJ and DD increased dramatically right after the start of the freeze-thaw cycles and there emerges no apparent nickpoints during the freeze-thaw cycles. Therefore, it can be concluded that one-year outdoor exposure has a negative effect on concrete frost resistance and the critical freeze-thaw cycles $N_f$ for DJ and DD specimens are regarded as 0 cycle. Moreover, there shows almost no obvious different between DJ and DD. Therefore, it is suggested that the different outdoor exposures DJ...
and DD have few influences on $N_f$. The mass moisture content of the specimens changing with time is exhibited in Fig. 4. As shown in the figure, compared with the peak point appearing at 54 cycles in the W/C 35%-1%-N specimen, the mass moisture content of DD and DJ specimens increased slightly until 8 cycles before it began to decrease continuously afterward. Table 4 exhibits the mass moisture content of W/C 35%-1% specimens with different curing conditions. As shown in the figure, the mass moisture content of W/C35%-1%-DJ at the critical freeze-thaw cycle $N_f$ is almost equivalent to the $M_{cr}$. However, for the W/C35%-1%-DD specimen, the mass moisture content at 0 cycles has exceeded the $M_{cr}$. W/C35%-1% concrete should appear significant damage right after the start of the freeze and thaw cycles, which also corresponds with the results by the length, RDM, and mass moisture content change. The DD condition is regarded as a more severe outdoor condition for W/C35%-1% specimens based on the fact that the length and mass moisture content change of 35%-1%-DD showing a higher value than that of the 35%-1%-DJ specimens. It is believed that the dry shrinkage during the summer in condition DD is stronger than in condition DJ. The dry shrinkage may help to result in more microcracks, which enhances the moisture absorption gradient during the freeze-thaw cycles.

The length change of 55%-1%-DJ and DD specimens are exhibited in Fig. 5. The length of 55%-1%-DJ and DD also increased much more rapidly than that of 55%-1%-N specimens. The one-year outdoor exposure also degraded W/C55%-1% concrete frost resistance. The mass moisture content change of 55%-1%-DJ and DD are exhibited in Fig. 6. Similar to W/C 55%-1%-N specimen, the mass moisture contents of DJ and DD decreased dramatically from the start of the cycles as well. Besides, the mass moisture contents at 0 cycle of 55%-1%-DJ and DD were inferior to that of 55%-1%-N specimen. It is reckoned that the W/C55%-1% specimens were damaged by freeze-thaw during the outdoor exposure before conducting the accelerated freeze-thaw test. Compared with the 55%-1%-N specimens, the frost damage was exacerbated during the outdoor exposure, and thus the mass moisture contents of 55%-1%-DJ and DD at the start of the cycles were below the W0 of 55%-1%-N. Furthermore, the mass moisture content of 55%-1%-DD at 0 cycle was less than that of 55%-1%-DJ specimen. The degradation by the curing condition DD is also severer in the W/C55%-1% specimens.

The length change of 55%-4.5%-DJ and DD specimens are illuminated in Fig. 7. The length change of the specimens in both DJ and DD conditions increased significantly until 88 cycles, while the nickpoint cycle for the 55%-4.5%-N specimens was 178 cycles. The one-year outdoor exposure also has an adverse impact on $N_f$ of W/C55%-4.5%. Fig. 8 illustrates the mass moisture content changes with freeze-thaw cycles. As shown in the figure, the nickpoint cycle in the mass moisture content curve of 55%-4.5%-DJ and DD specimens appeared at 88 and 64 cycles, respectively. Moreover, as shown in Table 4, the mass moisture content $W_{cr}$ at the nick point almost equals to the $M_{cr}$. Therefore, the outdoor exposure also shows a negative effect on W/C55%-4.5% concrete frost resistance.
4 Conclusion

This study investigates the relationship between the critical degree of saturation test and the accelerated freeze-thaw test by. A new concrete frost resistance criterion $N_f$ has been proposed. Furthermore, the effect of different outdoor exposures on concrete frost deterioration has been elucidated with by $N_f$. The primary conclusions are listed as follows.

1. Concrete own a critical mass moisture content $W_{cr}$ and it is not affected by the test methods. Once the critical moisture content is reached, concrete appears obvious deterioration under freeze-thaw exposure. The freeze-thaw cycle where $W_{cr}$ is
achieved is regarded as the critical freeze-thaw cycle $N_f$ and the $N_f$ is a new concrete frost resistance criterion.

2. The effect of different curing conditions on concrete frost resistance can be evaluated by $N_f$. Outdoor exposure has an adverse effect on concrete frost resistance. Besides, the condition that specimens set on the platform is a more severe condition for concrete frost deterioration.

References


Diagnosis of alkali aggregate reaction in concrete dams: an Indian case study

V. V. Arora, Brijesh Singh, Vikas Patel*, and B. N. Mohapatra

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Abstract: Deterioration of concrete structures have been observed in various dams in India and abroad. One of the critical deterioration mechanism involved in concrete hydraulic structures, such as dams and powerhouses can be attributed to the alkali aggregate reaction. Arch Dam in this case study is double curvature large thin arch dam, which features horizontal as well as vertical arches. Said dam suffered a continuous upstream deflection along with signs of minor distress. This paper provides a case study on systematic evaluation techniques adopted for diagnosis of Alkali Aggregate Reaction in Arch Dam in India. The study on arch dam constructed four decades ago in India included a field inspection of concrete dam structures with subsequent concrete cores drilling for laboratory investigation. The investigation included detailed petrographic evaluation of aggregates covering morphological microstructural and mineralogical analysis as per IS: 2386 Part VIII, mineralogical analysis covering the absence / presence of reactive aggregates prone to alkali-silica reaction, evaluation of concrete using petrographic analysis including study of pore structure and presence of micro cracks and abnormal reactive products, surface morphology study of concrete samples by Scanning Electron Microscopy (SEM) method and residual expansion on concrete cores.

Keywords: Alkali aggregate reaction, arch dam, petrographic analysis, scanning electron microscopy.

1 Introduction

Certain concrete aggregates react with the alkaline pore solution in concrete, which produces expansion leading to the cracking and deterioration of concrete. These reactions are known as alkali aggregate reactions (AAR). Among the first cases of AAR in India were a gravity dam and a power house built in the early sixties [1]. In an Australian dam, the occurrence of AAR was confirmed visually and with SEM [2]. XRD was additionally used. The first case identifying AAR in Switzerland reported in the literature is the Illsee dam [3]. Cracking of the concrete and displacements of up to 3 centimeters led to misalignments of machinery and difficulties to operate the installed equipment, like gates. Alkali-silica reaction is of more concern than alkali carbonate reaction because the occurrence of aggregates containing reactive silica minerals is more common. Alkali reactive carbonate aggregates have a specific composition whose occurrence is relatively rare [4,5]. Specifically with regard to the alkali aggregate reaction (AAR) due to the reactive aggregates, the study of this disease has been gaining increasing attention of the technical community due to several reports of this type of concrete deterioration. The AAR reaction leads to an expansion of the concrete and induce cracking and degradation of the mechanical properties. This implies problems in terms of serviceability, structural integrity [6,7,8] and durability since cracking favors the ingress of external species prone to initiate other degradations [9,10]. To deal with the affected structures, it is thus necessary to precisely understand the chemo-mechanical effects of each reaction. The potential for AAR in a large hydraulic structure should be thoroughly explored. Necessary measures should be taken to prevent or suppress the potential expansion within a tolerable limit.

AAR can significantly damage concrete structures. The gel produced by the reaction increases in volume by taking up water. This exerts an expansive pressure, causing unrestrained concrete to expand and restrained concrete to develop large compressive
forces. The rate of expansion caused by AAR typically has been found to be 20 to 200 \( \times 10^{-6} \) mm/mm/yr, depending on the severity of the reaction and the degree of restraint [11]. The volumetric increase can reach a maximum value of 0.2 to 0.5\% [12]. The expansion becomes detectable in about five to ten years after construction, and the most noticeable expansion may be detected in about 15 to 25 years. The expansion may continue for more than 50 years, although in some instances the expansion may cease after 20 to 30 years. The compressive stresses directly caused by AAR are usually within 3 to 4 MPa [11].

![Fig. 1 – Crack measurement on upstream and downstream side of upper gallery-3](image)

### Table 1 – Concrete mix design details

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Quantity – Mix Design (kg/m³)</th>
<th>Quantity – Actual Used (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement</td>
<td>245</td>
<td>327</td>
</tr>
<tr>
<td>Fly ash</td>
<td>43</td>
<td>0</td>
</tr>
<tr>
<td>Sand</td>
<td>511</td>
<td>447</td>
</tr>
<tr>
<td>4.75 – 10 mm</td>
<td>82</td>
<td>144</td>
</tr>
<tr>
<td>10 – 20 mm</td>
<td>163</td>
<td>165</td>
</tr>
<tr>
<td>20 – 40 mm</td>
<td>266</td>
<td>267</td>
</tr>
<tr>
<td>40 – 75 mm</td>
<td>348</td>
<td>347</td>
</tr>
<tr>
<td>75 – 150 mm</td>
<td>675</td>
<td>670</td>
</tr>
<tr>
<td>Water</td>
<td>118</td>
<td>121</td>
</tr>
<tr>
<td>Admixture</td>
<td>0.88%</td>
<td>%</td>
</tr>
</tbody>
</table>

Diagnosing AAR in this study included steps starting with the inspection of the structure. Based on the inspection and the available data, coring locations were defined and the samples taken were analyzed with different methods, starting with a visual assessment and finally extending to the residual expansion potential of concrete cores samples. The study on alkali silica reaction (ASR) is presented in this paper, in order to facilitate its identification and diagnosis in dams. Microscopy is emphasized as the major tool, as it makes possible to reliably identify even weakly developed ASR. In case of arch dam under investigation, the cracks were noticed in Gallery-3 on both upstream and downstream faces. These cracks were dispersed mainly in horizontal direction as shown in Figure 1. The width of cracks inside Gallery-3 on downstream face near rock and dam joint i.e. near abutment was more as compared to middle portion of Gallery-3. The crack width near abutment was around 0.45 mm and near central part of Gallery-3 was around 0.25 mm. The width of cracks in case of upstream part inside Gallery-3 was around 0.2 mm and was visible only in few portions.

During the construction OPC-33 grade cement was used. The two types of OPC-33 grade cement with 28 days strength of 38.83 N/mm² and 36.28 N/mm² were used. In the beginning, trial mix using available fly ash (Table-1) with cement OPC 33 grade were done but based on data available from the Quality Control Report of Dam during construction it has been found that use of fly ash was not adopted. The reason for not using fly ash as stated in the Quality Control Report of Dam is that fly ash was not conforming to the specification and there was large variation in the fineness of fly ash. The standard mix design adopted during the construction is given in Table-1. The alkali content of cement is a key property in the development of ASR and it should be determined for estimating the possibility of the reaction. Alkalinity of Portland clinker consist of sodium and potassium. Alkali content of cement can be determined as a \( \text{Na}_2\text{O} \) equivalent: \( \text{Na}_2\text{O} \) Equivalent \( (\text{Na}_2\text{O}\% + 0.658 \text{K}_2\text{O}\%) \). To avoid ASR \( \text{Na}_2\text{O} \) equivalent should be less than 0.60% according to standards. \( \text{Na}_2\text{O} \) equivalent in the used cement types varied between 0.80% and 1.35%. The alkali content...
is therefore can be considered sufficient for the development of ASR if aggregates are reactive. Though through petrography aggregates were found to be innocuous, aggregate expansion study was not carried out at the time of construction and if same was conducted the selection of pozzolanic cement might have been used in order to have better resistance to alkali aggregate reactivity. The second way out was to adopt of low-alkali cement, namely cement with an equivalent sodium oxide (Na₂O) content of 0.6% by mass or less to have better resistance to alkali aggregate reactivity.

2 Methods for Alkali Silica Reaction Identification

ASR is a reaction in concrete between the alkali hydroxides, which originate mainly from the Portland cement and certain types of aggregate. The ASR forms a gel that swells as it draws water from the surrounding cement paste. Reaction products from ASR have a great affinity for moisture. In absorbing water, these gels can induce pressure, expansion, and cracking of the aggregate and surrounding paste. To study ASR tests conducted includes visual inspection of concrete core samples, color test method for identifying concrete gels form by alkali silica reaction, petrographic analysis, XRD test, SEM, and residual expansion of concrete core samples.

2.1 Visual observation

Visual observation of all the accessible portions of three Galleries including upstream and downstream portion of dam were carried out at site and on the concrete core samples extracted from different locations. The visual inspections of concrete cores indicated rims indicating chances of ASR (Fig. 2). Thereafter, samples from rim portion were also sent XRD, Chemical analysis, SEM, and petrography studies.

2.2 Concrete core extraction for lab test and test performed

After the visual inspection of a dam, coring locations were identified. The choice was strongly influenced and limited by accessibility, usually more than in other structures. Survey data about the deformation of the dam is helpful in determining where expansion is relatively large or small, respectively. Special attention was given to areas where an expansion is critical for the integrity of the structure or where concentrations of stresses are likely to occur. The number of cores taken were based on the type and complexity of the dam under investigation and on the level of detail and representativeness required. Concrete cores of 150 mm and 100 mm diameter were extracted from all three galleries (inside and outside), downstream bottom and upstream side of dam. Total about sixty concrete cores were extracted covering entire dam structure. The tests included evaluation of aggregates (taken out from concrete core) using petrographic analysis including morphological microstructural and mineralogical analysis as per IS: 2386 Part VIII (1963) [19], detailed mineralogical analysis covering the absence/presence of reactive aggregates prone to alkali-silica reaction, evaluation of concrete using petrographic analysis including study of pore structure and presence of micro cracks and abnormal reactive products, surface morphology study of concrete samples by SEM including detection of fracture pattern and residual expansion of concrete core samples.

<table>
<thead>
<tr>
<th>Location</th>
<th>pH</th>
<th>SO₄²⁻ mg/l</th>
<th>Cl⁻ mg/l</th>
<th>Inorganic</th>
<th>Organic</th>
<th>Suspended</th>
<th>Solids (mg/l)</th>
<th>Alkalinity (mL)</th>
<th>Acidity (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Downstream</td>
<td>6.59</td>
<td>34.30</td>
<td>10.76</td>
<td>32.00</td>
<td>9.00</td>
<td>18.00</td>
<td>5.90</td>
<td>0.80</td>
<td></td>
</tr>
<tr>
<td>Upstream</td>
<td>6.78</td>
<td>15.43</td>
<td>16.14</td>
<td>20.00</td>
<td>4.00</td>
<td>8.00</td>
<td>2.30</td>
<td></td>
<td>0.40</td>
</tr>
<tr>
<td>Gallery-1</td>
<td>7.56</td>
<td>42.18</td>
<td>16.14</td>
<td>102.00</td>
<td>54.00</td>
<td>16.00</td>
<td>19.00</td>
<td></td>
<td>Nil</td>
</tr>
<tr>
<td>Gallery-2</td>
<td>6.92</td>
<td>40.47</td>
<td>23.32</td>
<td>612.00</td>
<td>66.00</td>
<td>33.00</td>
<td>70.00</td>
<td></td>
<td>1.20</td>
</tr>
<tr>
<td>Gallery-2 Drain</td>
<td>6.33</td>
<td>28.81</td>
<td>16.14</td>
<td>22.00</td>
<td>6.00</td>
<td>4.00</td>
<td>1.80</td>
<td></td>
<td>0.80</td>
</tr>
<tr>
<td>Gallery-3</td>
<td>7.38</td>
<td>37.38</td>
<td>17.94</td>
<td>92.00</td>
<td>10.00</td>
<td>7.00</td>
<td>11.00</td>
<td></td>
<td>0.40</td>
</tr>
</tbody>
</table>

| Limiting Value for RCC works as per IS:456-2000 | >6 | 400 | 500 | 3000 | 200 | 2000 | 25 | 5 |

Fig. 2 – ASR rims from concrete cores extracted from different locations
2.3 Chemical analysis of concrete samples of dam
The chloride, sulphate, pH and other chemical parameters are within the permissible limit given in IS: 456 (2000) [19] for both dams. No adverse chemical presence in concrete and water samples in general is seen in Table 2. The SO3 content calculated from chemical analysis of concrete core samples in case of arch dam varied from 0.30 to 0.50% per m3 of concrete and Na2O equivalent (water soluble acid method) in concrete of arch dam varied from 0.16 to 0.30 per m3 of concrete.

2.4 Mineralogical studies in arch dam
The petrographic analysis was done for evaluation of aggregates (taken out from core). The study included Petrographic and Mineralogical analysis of the samples as per IS: 2386 Part VIII [19]. Mineralogical details were analyzed covering the absence/presence of reactive aggregates prone to Alkali-Silica reaction. Thin sections of the selected samples were prepared. The samples were studied in NIKON POL-600E microscope under polarized light. The modal analysis, granulometry and microstructures were done using the Image Analysis System attached with the microscope. The petrographic study of aggregate taken from concrete core samples extracted randomly from entire dam indicated aggregate type as Hypersthene-Granite (Fig. 3). The major mineral constituents were orthoclase-feldspar, quartz, hypersthene and plagioclase-feldspar. Accessory minerals were pyrite, microcline-feldspar & iron oxide. Grain size of quartz varied from 18 µm to 478 µm with an average of 264 µm. Majority of quartz grains were in the size range of 200 µm to 260 µm. The strained quartz percentage is about 16% and their undulatory extinction angle (UEA) varied from 190 to 210. Lath shaped hypersthene grains were partially altered. The modal composition obtained was: (a) Trade Group: Granite (Igneous Rock), (b) Petrological name: Hypersthene-Granite, (c) Particle shape: Irregular and (d) Surface texture: Crystalline. Based on petrographic studies, it was also observed that, orthoclase grains present in coarse aggregate were affected more than other feldspar. However, alterations of minerals were not very common hence petrographically it is concluded that aggregates were partially affected by hydration reactions and their hydration products. The petrography analysis of concrete samples indicated presence of onset of ASR (preliminary stage) and examination of ASR rims indicated that the infection was due to presence of orthoclase.

The detection of Alkali Silica Reaction swelling in concrete by staining method (color test) was done based on method proposed by GD Guthrie et.al [13]. The study included the sequential application of solutions of each of two water soluble compounds. Concentrated solutions of sodium cobaltinitrite and rhodamine B in water are prepared. The concrete surface to be examined was treated by pre-rinsing with water and subsequently applying each solution to the surface. After 30-60 seconds, the concrete was rinsed thoroughly with water. The treated surface will show yellow and pink regions where ASR gel is present, yellow regions indicate the presence of K-rich, Na-K-Ca-Si gels. While pink regions indicate alkali-poor gels. The final rinse step was required, since the yellow sodium cobaltinitrite solution will coat the entire concrete surface as will the pink rhodamine B solution, thereby obscuring the stained gel regions. Best results were obtained when the sample is treated first with the sodium cobaltinitrite solution; however, the application of the rhodamine B solution first gave adequate results (Fig. 4).

The slices of core samples were rinsed with water and rinsed surfaces were searched for regions of yellow staining and regions of pink staining whereby K-rich i.e. Na-K-Ca-Si gels generated from ASR were identified by yellow staining and alkali poor, Ca-Si generated from ASR were identified by pink staining. The test results indicated light to dark pinkish color in all cases indicating presence of ASR.

Table 3 – Modal composition of the coarse aggregate (results in %)

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Sample No.</th>
<th>Rock type</th>
<th>Minerals</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Orthoclase-feldspar</td>
</tr>
<tr>
<td>1</td>
<td>Coarse aggregate</td>
<td>Hypersthene-granite</td>
<td>35</td>
</tr>
</tbody>
</table>
2.5 Scanning electron microscopy studies

The most widespread tool to characterize the microstructure and the local chemical composition of cement-based materials is the SEM. Samples for SEM were selected, dried and impregnated. After removing 1-2 mm by polishing to avoid artifacts introduced by cutting and grinding the sample before impregnation, the surface to-be-investigated were polished. The concrete core samples were examined under SEM and based on study carried out it was seen that numerous microcracks were observed at the interfacial zone and in the paste (Fig. 5). Ettringite formation of size ranging less than 2 microns to 60 microns was observed in most of the samples. Pyrite (FeS$_2$) crystals were present as minor constituents in Hypersthene granite (up to 1-4%). Microscopic studies revealed that pyrite was mainly outsourcing sulphur for formation of ettringite which was randomly distributed in the coarse aggregate. To ascertain the ettringite formation in terms of percentage, ten samples from each concrete core were taken under observation. The studies were carried out with 1000 counts from each core. The percentages of ettringite formation vary from less than 1% to 3% of 5% to 8% of open air voids present in the concrete. The ettringite formation of order 1% to 3% is not likely to cause any expansion.

When pores were studied under microscope, it was observed that ettringite formation had taken place with three types i.e. crystalline, semi-crystalline and gel (Fig. 6).

Due to the ageing effect on concrete, the three types of feldspars and pyrite present in the coarse aggregate may lead to disintegration and transformation into either in other mineral or by-product. However, this disintegration would most likely get deposited in-situ. Under conditions of extreme deterioration and repeated wetting and drying, ettringite crystals can appear to completely fill voids or cracks. However, ettringite, found in this benign state as large needle-like crystals, should not be interpreted as causing the expansion of deteriorating concrete (PCA R&D 2002) [14].
2.6 X-Ray Diffraction (XRD) Analysis

The XRD test was carried out on the concrete core samples to determine their mineral composition. XRD is based on principle of diffraction of X-rays by atomic planes of the material. When monochromatic X-ray beam falls on the crystalline material the scattered X-rays from the atomic planes get interfere with each other to produce constructive or destructive interference depending on the inter atomic planes distance.

\[ 2d \sin \Phi = n\lambda \]  

where \( d \) = inter planar distance [Å], \( \lambda \) = wavelength [Å], \( \Phi \) = angle by the incoming beam with the normal of the plane, and \( n \) = order of diffraction.

If Bragg’s law is satisfied, it results in constructive interference. The basic parts of XRD include X-ray tube, Sample Holder, Gonio meter and Detector. X-ray tube generator the X-rays by the interaction of fast moving electrons with target metal & generated X-rays are made palled using soller slits & allow to fall on sample. The diffracted X-rays from the sample are detected by detection system. Gonio meter allows the measurement at different angles for e.g. from 00 to 650. The recorded intensity pattern over range of 20 is processed for phase identification using software. The X-Ray Diffraction (XRD) Analysis is conducted on concrete core samples from all the ten locations. The each concrete core sample is analyzed for aggregate, mortar and concrete part. The typical results of XRD test is given in Table 4. The X-Ray Diffraction (XRD) analysis was conducted on powder obtained from concrete cores and based on test results, it is seen that there is no significant unhydrated part left in the samples and no extraordinary phases are identified. The sulphate was found in the form of either Pyrite or Chalcopyrite. Minor ettringite formation is also detected. ASR and thaumasite were also identified in the few samples under SEM study. ASR rims were also checked for Alkali Silica Reaction products. The results obtained revealed that initial stage of ASR was observed on the boundaries between coarse aggregate and cement mortar. Aggravated ASR reactions
were more on the boundaries between partially altered coarse aggregate used and mortar part. The thickness of ASR rims varies from few microns to 50 microns in size. In few instances, the pores containing crystalline mass were observed on the boundaries of ASR rims. Microcline grains were also partially affected by ASR but effect of ASR on Microcline was less aggressive than orthoclase. In few instances, some plagioclase grains had also shown effect of ASR on the grain boundaries.

2.7 Residual expansion test on concrete cores and accelerated mortar bar test on aggregates

Accelerated mortar bar test as per ASTM C-1260 (2014) [15] for evaluating residual expansion of coarse aggregate was carried out on coarse aggregate samples taken from concrete cores. For this purpose, the aggregates removed from the concrete cores were fragmented into artificial sand to obtain the necessary fractions to cast mortar bars measuring 25 mm x 25 mm x 285 mm with a standard cement by proportioning one part of cement to 2.25 parts of graded aggregates by mass, a fixed w/c ratio of 0.47. The sample after 24-hours was demolded and then cured in hot water at 80°C for 24-hours. Finally, the specimen is stored in 1N NaOH solutions at 80°C for 14 days. The length change observations were taken in hot condition (within 20 seconds after taking out from the solution). The samples were stored in plastic containers and the use of glass or metal container for this purpose was not recommended as the same get corroded by NaOH solution. As per ASTM criteria, the aggregate showing 14 days expansion less than 0.10% are classified as innocuous (non-reactive), whereas the aggregates showing more than 0.20% expansion are classified as potentially reactive. For aggregates showing expansion between 0.10% and 0.20%, the aggregates reactivity is classified as inconclusive and the results are to be supported by another test. The test results of accelerated Mortar Bar Test as per ASTM C-1260 (2014) [15] indicated that the net expansion in coarse aggregate sample is 0.04%. However, since the coarse aggregate seems to be non-reactive, there is possibility of fine aggregate being reactive.

The 14 days test results of accelerated Mortar Bar Test conducted on concrete core of 150 mm diameter and 280 mm length in similar lines to ASTM C-1260 (2014) [15] for determining residual expansion indicated the residual expansion of 0.17%. As the residual expansion of concrete samples even after the age of 40 years are between 0.10 to 0.20 there is possibility of aggregate being slow reactive though not under potentially reactive category. To cross-check this, the concrete core specimens from both wet and dry portions of dam were selected for residual expansion testing in similar line to procedure in ASTM C 1293 (2018) [16] to determine the susceptibility of a concrete to the alkali-silica reaction. The concrete core was tested with deviation that temperature was changed from 38°C to 60°C. The change in temperature was done from 38°C to 60°C because it was seen in the past studies done by NCB that the slow reactive aggregate gives expansion at 60°C. The higher temperature has been also recommended in IS: 2386 Part VII (1963) [18] and IS: 383 (2016) [20] also. The reading was taken up to one year. An average expansion was calculated from measurements on three replicate specimens. If the average expansion of the three concrete bars is equal to or greater than 0.04% at an age of one year, then the aggregate is considered to be potentially reactive. The humidity level of 100% was maintained during testing. The average residual expansion of concrete cores in case of arch dam varied from 0.0392 to 0.0396% (Fig. 7). This average residual expansion at more than 40 years age is significant keeping in view that the limit of 0.04% at the age of one year is for one year old concrete and this indicates that there may be the chance of slow ASR. The same was also indicated in preliminary stage by SEM and Petrography studies.

Table 4 – X-ray diffraction (XRD) analysis of concrete sample

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Location</th>
<th>Findings from X-Ray Diffraction (XRD) Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Downstream</td>
<td>Minor Sulphates in the form of Chalcopyrites and Pyrites. Major Portlandite and Minor Calcite. Minor Ettringite</td>
</tr>
<tr>
<td>2</td>
<td>Upstream</td>
<td>Minor Sulphates in the form of pyrites and Chalcopyrites. Major Portlandite and Minor Calcite (CaCO3). No significant unhydrated part left and no extraordinary phases are identified. Minor Ettringite</td>
</tr>
<tr>
<td>3</td>
<td>Gallery-3</td>
<td>Minor Portlandite and Sulphates in the form of pyrite and Chalcopyrite. Minor Ettringite and Minor Calcite. No significant unhydrated part left in the sample and no extraordinary phases are identified</td>
</tr>
</tbody>
</table>

The study on expansion potential of aggregates expansion indicates that the aggregate may not be under potentially reactive category and the typical pattern cracking due to ASR will not occur but length change can be caused even by small amount of ASR expansion. With the end restraints in case of arch dam, this small expansion has contributed to the movement of mid-point of dam towards upstream.
Results and Discussion

From the study done, it can be seen that even though there was no visual impression of Alkali Silica Reaction evident. However the detailed investigation covering visual inspection of inside concrete by coring, color test method for detection of ASR, petrographic analysis for evaluation of aggregate type and its mineral composition, X-Ray diffraction and SEM study including the residual expansion of aged concrete all together needs to be done for proper understanding of distress mechanism due to ASR. In the current study, the ettringite growth was noticed along with ASR but ettringite percentage was very less and literature study also indicates that the ettringite in the benign stage may not be the cause of distress in dam. No adverse chemical presence in concrete and water samples in general is seen. However, alkali content in concrete by water soluble acid method is high and this high alkali may cause some ASR reaction even if aggregate is moderate reactive. The petrographic analysis of coarse aggregate indicates aggregate type as Hypersthene-Granite. The strained quartz percentage and their UEA are within permissible limits. Feldspar grains are partially fractured, shattered and altered. Reaction rims are developed mostly on the margins of the euhedral pyrite grains. In few instances pyrite grains are completely consumed during hydration reaction. The Petrography analysis of concrete samples indicates presence of onset of Alkali Silica reaction (preliminary stage). The slice of concrete core samples were rinsed with water and rinsed surfaces were searched for regions of yellow staining and regions of pink staining whereby K-rich i.e. Na-K-Ca-Si gels generated from ASR were identified by yellow staining and alkali poor, Ca-Si generated from ASR were identified by pink staining. The test results indicated light to dark pinkish colour in all cases indicating presence of ASR. The sulphate in the form of pyrite and Chalcopyrite is found from petrographic, SEM and XRD studies and this is the reason for ettringite formation. The percentages of ettringite formation vary from less than 1 percent to 3 percent. The ettringite formation of order 1 percent to 3 percent is not likely to cause any expansion. Based on the test results of XRD samples from all the locations, it is seen that there is no significant unhydrated part left in the samples and no extraordinary phases are identified. The sulphate in the samples is found in the form of either Pyrite or Chalcopyrite in aggregates. Minor ettringite and ASR formation is also detected in few cases from XRD test results.

ASR though seen in the preliminary stage and validated through residual expansion test may cause expansion in dam keeping in view that the dam is restrained from both ends as the 14 days test results of accelerated Mortar Bar Test conducted on concrete core indicated the residual expansion of 0.17%. As the residual expansion of concrete samples even after the age of 40 years are between 0.10 to 0.20 there is possibility of aggregate being slow reactive though not under potentially reactive category. The average residual expansion of concrete cores by long term test varied from 0.0392 to 0.0396% indicating that the average residual expansion at more than 40 years age is significant keeping in view that the limit of 0.04 % at the age of one year is for one year old concrete and this indicates that there may be the chance of slow Alkali Silica Reaction. With the end restraints in case of arch dam, this small expansion has contributed to the movement of mid-point of dam towards upstream side. Keeping in view the support conditions and shape of dam structure there is need to review the limits of ASR test value of aggregate to be used in Arch dam.

Conclusions

Diagnosis of Alkali Silica Reaction in aged concrete dams is important for deciding measures to prevent further distress in the hydraulic structures such
as dams. The detailed investigation of dam covering visual inspection of inside concrete by coring, colour test method for detection of ASR, petrographic analysis for evaluation of aggregate type and its mineral composition, X- Ray diffraction and SEM study including the residual expansion of aged concrete all together is essential for proper understanding of distress mechanism due to ASR. The study also indicated that at the small amount of expansion due to slow reactive aggregates can be critical in Arch dam considering the end restraints. The study indicated the simultaneous occurrence of ettringite formation in the dam but the percentage of ettringite formation was less than 1-3% of 5-8% of open air voids. The ettringite in such small quantum may not be interpreted as cause of expansion. The results of Accelerated Mortar Bar Test conducted on cores in similar lines to ASTM C-1260 (2014) for determining residual expansion indicated the net expansion 0.17%. The average residual expansion of concrete cores in similar to procedure of ASTM C-1293 (2018) varied from 0.0392 to 0.0396%. This average residual expansion at more than 40 years is significant keeping in view that the limit is 0.04% at the age of one year for one year old concrete and this indicates that there may be the chance of slow ASR. The expansion study indicates that though the aggregate is not under potentially reactive category and the typical pattern cracking due to ASR will not occur but length change can be caused even by small amount of ASR expansion. With the end restraints in case of arch dam, this small expansion may also add to the movement of mid-point towards upstream side. Keeping in view the support conditions and shape of dam structure there is need to review the limits of ASR test value of aggregate to be used in arch dam.

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The authors would like to acknowledge the NCB laboratory technical officers and technical assistants, Central Project Management Unit DRIP, Central Water Commission-India, Kerala State Electricity Board (KSEB) as well as World Bank for their assistance in carrying out this sponsored project.

References

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Deflection hardening behavior and elastic modulus of one-part hybrid fiber-reinforced geopolymer composites

Yazan Alrefaei, and Jian-Guo Dai

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Abstract: This paper reports for the first time the experimentally tested elastic modulus values of the fiber-reinforced geopolymer composites (FRGCs). Further, the deflection hardening behavior and sand addition effect on the ambient-cured one-part steel-polyethylene FRGCs are also reported wherein the total fiber volume fraction used was 2%.

It was found that although the cylinders’ compressive strength of the blended-based FRGCs was relatively higher than that of the slag-based FRGCs, the latter showed a relatively larger elastic modulus in comparison with the former. The addition of fiber increased the elastic modulus of the geopolymer composites relative to the none-fibrous composites at which the elastic modulus improvement was related to the steel (ST) volume. Further, all the FRGCs exhibited a deflection hardening behavior which was directly related to the polyethylene (PE) volume included in the composite. Generally, the slag-based FRGCs showed better flexural behavior (i.e. modulus of rupture, deflection capacity, and multiple-cracking behavior) in reference to the blended-based FRGCs. The sand addition (i.e. small size like 212 µm with low content such as 30% by mass) was found to improve both flexural cracking and ultimate strengths without affecting the deflection capacity nor the cracking behavior. This could be due to the better volume stability and lower shrinkage of the geopolymer matrix after sand addition.

Keywords: Alkali-activated materials; Fly ash; Slag; Strain hardening; Multiple cracking; Elastic modulus.

1. Introduction

Fiber reinforced cementitious composites (FRC-Cs) have been studied extensively in the past decades and yet are still being researched and developed up to date. FRCC is the general terminology used to describe all fibrous cementitious composites in which a subgroup named ductile fiber-reinforced cementitious composites (DFRCC) was developed later on. DFRCC terminology was used to describe the composites that exhibit a multiple-cracking behavior in flexure only. A special class of DFRCCs termed as High-Performance fiber reinforced cementitious composites (HPFRCC) was formed to represent the composites that attain strain hardening and multiple cracking in both tension and flexure. Such classification was introduced by the DFRCC committee in the Japan Concrete Institute (JCI) [1].

One of the commonly known examples of HPFRCCs is the engineered cementitious composites (ECC) that include about 2% fiber content (by volume) and can attain a strain capacity of up to 8% [2, 3]. Thanks to such high ductility and strength, the ECCs are proposed for several structural implementations [4, 5]. However, in order to manufacture such high-ductile ECCs, a relatively large volume of cement should be used, up to 2-3 times that required for concrete production [6]. Accordingly, the sustainability and eco-friendliness performances of ECCs will be concerning as the cement production is responsible for 5-7% of the total CO₂ emissions worldwide [7]. Therefore, the researchers headed to implement the idea of clinker-free cement-less geopolymer binders in FRCCs to develop a new class of green materials called “Fiber Reinforced Geopolymer Composites” (FRGs) and “Engineered Geopolymer Composites” (EGCs).

Despite the fact that the FRGCs’ research area is reasonably new [8], many studies were conducted on the tensile behavior of FRGCs reinforced with several types of either mono [9-15] or hybrid [16-19]
fibers (i.e. basalt, carbon, cotton, glass, polypropylene (PP), polyvinyl alcohol (PVA), polyethylene (PE) and steel) and synthesized using different precursors (i.e. fly ash-based, slag-based and blended-based). Further, limited studies also investigated the flexural behavior of the FRGCs whether reinforced with mono-fibers [8, 18, 20-22] or hybrid-fibers [17, 19, 23, 24]. On the other hand, the elastic modulus (E) of the FRGCs was barely tested in experiments according to previous literature [17]; yet, part of the research reported the theoretical E calculated based on the matrix fracture properties [12, 16, 21, 25, 26].

Generally, the production of geopolymer binders can follow two terminologies either “one-part” or “two-part” [27-29]. The two-part terminology requires the application of solution type activators to activate the aluminosilicate precursors, while the one-part or “just add water” terminology implements the use of solid activators that are pre-mixed with the precursors before the addition of water. Due to the relatively high corrosiveness and hostility of the alkali activator solutions [30], it is predicted that the implementation of two-part geopolymer binders will prosper in the precast concrete industry where the casting environments in factories are more controllable. On the other hand, the one-part geopolymer binders will provide an easy toolkit alternative to ordinary Portland cement (OPC) for in-situ applications; yet such methodology is still under research. According to previous research [27, 31], the commercial anhydrous sodium metasilicate is the most commonly used and efficient solid activator in the industry of one-part geopolymer binders.

Overall, the bulk of the studies mentioned in the literature followed the terminology of two-part geopolymer, while limited studies dealt with the one-part FRGCs [16, 25, 32]. Further, the research related to ambient-cured FRGC focused on the blended-based geopolymer [17, 18, 20, 23, 33] (i.e. fly ash-slag combinations with different ratios), however finite papers investigated the slag-based geopolymer behavior when incorporated with fibers [32]. Additionally, some studies focused on the fiber hybridization concept [17, 22-24, 34] including steel, PP and PVA fibers, yet no study was conducted on the hybrid combination of straight steel fiber and PE. Hence, there is a short in the research related to the flexural behavior and elastic modulus of FRGCs, especially when incorporated with hybrid-fibers and one-part geopolymer concepts.

The aim of this study is to better understand the effect of combining the concepts of hybrid-fibers and one-part geopolymer on the mechanical behavior of FRGCs to permit the structural implementations of FRGCs as green repairing materials. Such research will contribute to the ongoing research related to FRGCs by extending the available database results.

2. Theoretical Criteria for Deflection Hardening Behavior

As shown in Fig. 1, the typical deflection hardening behavior is mainly characterized by two points called limit of proportion (LOP) and modulus of rupture (MOR). According to ASTM C1018 [35], the LOP point is defined as the point where the load-deflection curve nonlinearity becomes conspicuous; in other words, such point represents the first cracking load. However, the identification of such a point can be challenging in the fibrous composites exhibiting deflection hardening behavior; thus, the method proposed by Kim et al. [36] was followed in this study. The MOR point, or else known as the ultimate flexural strength, is defined as the point following the LOP where the softening starts to occur afterward. \( P_{LOP} \) and \( P_{MOR} \) are representing the cracking and ultimate loads, respectively; while their corresponding deflections are labeled as \( \delta_{LOP} \) and \( \delta_{MOR} \), respectively, as shown in Fig. 1.

Two conditions shall be satisfied in order to assure the deflection hardening behavior of a composite. First, the ultimate load \( (P_{MOR}) \) must be larger than the cracking load \( (P_{LOP}) \). Second, the deflection at ultimate \( (\delta_{MOR}) \) needs to exceed that at first cracking \( (\delta_{LOP}) \). The higher the gap between the cracking and ultimate loads and deflections, the better deflection hardening behavior and ductility can be achieved. Accordingly, this concept can be interpreted into a new value called the “ductility index (DI)” which is the ratio of \( \delta_{MOR} \) to \( \delta_{LOP} \). The ductility of a composite is directly related to its DI value; in other words, the higher the DI value is, the more ductile the composite will be.

3. Materials

For geopolymer matrix preparation, local Hong Kong low-calcium Class-F fly ash (FA) and...
ground granulated blast-furnace slag (GGBS) imported from mainland China were used in this study. The chemical compositions of the precursor materials are reported in Table 1, as determined by X-ray Fluorescence (XRF) test. Fig. 2 shows the morphologies of the raw precursor materials using scanning electron microscope (SEM) wherein the FA particles were spherical in shape with a relatively high content of irregular shaped glassy contents, while the GGBFS particles were mainly anomalous. Figs. 3 a and b display the X-ray Diffraction (XRD) patterns and the particle size distribution of the raw precursor materials, respectively. Clearly, the FA contained relatively high crystalline content while relatively large amorphous hump was noticed in the GGBS XRD pattern. The GGBS particle size was relatively larger than that of FA particles where the d_{50} values of GGBS and FA were 13 and 24 microns, respectively.

Table 1 – Chemical composition of fly ash and slag determined by XRF

<table>
<thead>
<tr>
<th>Parameters</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>Fe₂O₃</th>
<th>MgO</th>
<th>SO₃</th>
<th>TiO₂</th>
<th>K₂O</th>
<th>P₂O₅</th>
<th>SrO</th>
<th>LOI^a</th>
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<tbody>
<tr>
<td>FA</td>
<td>44.4</td>
<td>32.6</td>
<td>6.67</td>
<td>6.49</td>
<td>1.86</td>
<td>2.27</td>
<td>1.24</td>
<td>1.81</td>
<td>0.44</td>
<td>0.14</td>
<td>5.55</td>
</tr>
<tr>
<td>GGBS</td>
<td>18.9</td>
<td>6.43</td>
<td>66.9</td>
<td>0.74</td>
<td>1.41</td>
<td>1.97</td>
<td>1.88</td>
<td>0.67</td>
<td>0.08</td>
<td>0.18</td>
<td>0.25</td>
</tr>
</tbody>
</table>

^a Loss on ignition

Fig. 2 – The morphology of raw precursor materials: a) FA and b) GGBS

Fig. 3 – Raw precursor materials properties: a) XRD patterns and b) particle size distribution
Table 2– Properties of fibers

<table>
<thead>
<tr>
<th>Fiber type</th>
<th>Length (mm)</th>
<th>Diameter (µm)</th>
<th>Modulus of elasticity (GPa)</th>
<th>Strength (MPa)</th>
<th>Density (g/cm³)</th>
</tr>
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<tr>
<td>ST</td>
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<td>180</td>
<td>200</td>
<td>2850</td>
<td>7.8</td>
</tr>
<tr>
<td>PE</td>
<td>13</td>
<td>17</td>
<td>114</td>
<td>3000</td>
<td>0.97</td>
</tr>
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</table>

Table 3– Mix proportions (by wt.) and fiber content

<table>
<thead>
<tr>
<th>Series</th>
<th>Mix ID</th>
<th>Hybridization %</th>
<th>Binder</th>
<th>Activator /Binder</th>
<th>Water /Binder</th>
<th>Sand /Binder</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>PE</td>
<td>ST</td>
<td>Slag</td>
<td>Fly ash</td>
<td>0.12</td>
</tr>
<tr>
<td>1</td>
<td>P: FRGC-S</td>
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<td>0.0%</td>
<td>1</td>
<td>-</td>
<td>0.12a</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.5%</td>
<td>0.5%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.0%</td>
<td>1.0%</td>
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<tr>
<td></td>
<td></td>
<td>0.5%</td>
<td>1.5%</td>
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<td></td>
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<tr>
<td></td>
<td></td>
<td>0.0%</td>
<td>2.0%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>P: FRGC-FA/S</td>
<td>2.0%</td>
<td>0.0%</td>
<td>0.5</td>
<td>0.5</td>
<td>0.12a</td>
</tr>
<tr>
<td></td>
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<td>0.5%</td>
<td></td>
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<td>1.0%</td>
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<td>2.0%</td>
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</tr>
<tr>
<td>3</td>
<td>M: FRGC-S</td>
<td>1.5%</td>
<td>0.5%</td>
<td>1</td>
<td>-</td>
<td>0.12a</td>
</tr>
<tr>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

a anhydrous sodium metasilicate powder
b with a maximum size of 212 µm

In order to produce the one-part geopolymer matrix, the solid alkali activator used was anhydrous sodium metasilicate powder based on the recommendation of Nematollahi, et al. [31]. The anhydrous sodium metasilicate powder composed of 50.46% Na₂O and 47.24% SiO₂ by weight; accordingly, the modulus ratio (Ms) of such activator was 0.94 (where Ms = SiO₂/Na₂O).

The fibers used in this study were copper-coated straight-shaped steel fibers (ST) and ultra-high molecular weight polyethylene fibers (PE). Table 2 presents the mechanical properties of both fibers as provided by the manufacturers.

Fine silica sand with a maximum size of 212 µm imported from mainland China was used in this study which was adapted from Nematollahi, et al. [26]. It is good to mention that such small sand size was chosen to minimize the fracture toughness and first cracking strength of the geopolymer matrix which is beneficial for the deflection hardening behavior of the composite [16].

4. Experimental Program

The deflection hardening behavior and the elastic modulus of one-part steel-polyethylene FRGCs were experimentally investigated in this study. The effects of different hybridization ratios, different precursor materials (e.g. 100% GGBS matrix and 50/50 FA/GGBS matrix) and sand addition were also highlighted.

4.1 Mix proportions, procedure, casting and curing

The experimental mix proportions and fiber contents followed in this paper were similar to those mentioned in Alrefaei and Dai [16] as shown in Table 3. The precursors used to produce the FRGCs were 100% GGBS and a blend of 50% fly ash with 50% GGBS in Series 1 and 2, respectively. Five different hybrid combinations ranging between 0% to 2% of ST and PE fibers were used in both Series 1 and 2 while the total fiber volume fraction was kept at 2%. Series 3 included the hybrid composite of 1.5% PE and 0.5% ST with both slag-based and blended-based geopolymer matrix in addition to the 212 µm fine sand. As shown in Table 3, the sand and the activator contents used in this study were 30% and 12%, respectively, resulting in an alkali concentration (Na₂O % by the mass of binder) of 6%. The sand content was adapted from Nematollahi, et al. [26] while the activator content was recommended by Alrefaei and Dai [16]. Tap water was used in all the mixes and the sand was oven-dried before usage. The water-to-binder ratio used in all FRGC mixes was 0.45 which was equivalent to a liquid-to-solid ratio of 0.4 with the aim of guaranteeing good workability and better fiber dispersion in the composites.
The codified mix ID includes three alphanumeric character parts at which the first part mentions the geopolymer matrix: “P” for paste and “M” for mortar, the second part symbolizes the fiber-reinforced geopolymer composite shortcut “FRGC” and finally the third part specifies the precursor used to synthesize the geopolymer matrix: “S” for slag and “FA/S” for blended geopolymer composites.

Fig. 4 – Schematic of bending test set-up

Four sheet specimens in addition to four cylinders were cast for each FRGC mix. The sheet specimens were 300 mm, 75 mm and 20 mm in length, width, and thickness, respectively. The cylinders were 50 mm and 150 mm in diameter and height, respectively. Hobart mixer was used to prepare all the FRGC within 15 to 20 minutes duration. The mixing procedure of geopolymer matrix and fiber addition techniques were adapted from Alrefaei & Dai [16] and Alrefaei, et al. [37], respectively. All the specimens were prepared and cast using a single batch. A mechanical vibrating table was used to compact the FRGC samples following Alrefaei, et al. recommendations [37] wherein mono-steel composites were not over vibrated to prevent fiber segregation. To avoid drying shrinkage cracks, all the specimens were covered with wet burlap and plastic sheets for 24 hours after casting. The specimens were demolded after 24 hours then placed in a water tank for the next 27 days. All the tests were conducted at the age of 28 days.

4.2 Instrumentation and testing procedures

Four points bending test was used to study the deflection hardening behavior of the achieved FRGCs. The schematic of the bending test set-up and sheet specimen dimensions are shown in Fig. 4. A universal testing machine was used to test the sheet specimens with an extension control rate of 0.5 mm per minute [22]. A 50 mm capacity LVDT was used to measure the mid-span deflection during the bending test. The flexural strength was calculated using the following equation [38]:

\[
\sigma = \frac{3P(L-L_i)}{2bd^2} \quad \text{………………… (1)}
\]

where \(\sigma\) is flexural stress; \(P\) is the load; \(L\) is the length of the support span (260 mm); \(L_i\) is the length of the loading span (100 mm); \(b\) is the width (75 mm) and \(d\) is thickness (20 mm) of the sample. The ductility index (DI) was calculated using the following equation [22]:

\[
DI = \frac{\delta_{MOR}}{\delta_{LOP}} \quad \text{………………… (2)}
\]

where \(\delta_{MOR}\) is the deflection at ultimate peak load and \(\delta_{LOP}\) is the deflection at first cracking load.

The cylinders were tested in compression using a hydraulic testing machine with an extension control rate of 0.03 mm per minute in accordance with ASTM C39 [39], while the elastic modulus was calculated using the equation mentioned in ASTM C469 [40]. The change of the cylinder’s length was measured over a gauge length of 100 mm. All the samples were capped using gypsum in compliance with ASTM C617 [41]. The cylinder test setup is shown in Fig. 5.

Fig. 5 – Test set-up of cylinder compression

5. Results, Analysis, and Discussion
All experimental test results (i.e. Average ± Standard Deviation) are briefed in Table 4. The discussion and analysis of the results will be carried out in detail in the following sections. The error bars were not included in some figures in this paper to maintain clarity.

Table 4 – Summary of test results

<table>
<thead>
<tr>
<th>Series</th>
<th>Mix ID</th>
<th>Fiber content (%)</th>
<th>$s_{LOP}$ (MPa)</th>
<th>$\delta_{LOP}$ (mm)</th>
<th>$s_{MOR}$ (MPa)</th>
<th>$\delta_{MOR}$ (mm)</th>
<th>DI</th>
<th>No. of cracks</th>
<th>$f_{cu}$ (MPa)</th>
<th>$f_{cm}$ (MPa)</th>
<th>$E$ (GPa)</th>
<th>$E_{m}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>P: FRGC-S</td>
<td>2.0%</td>
<td>0.0%</td>
<td>2.7±0.4</td>
<td>1.0±0.2</td>
<td>8.2±1.7</td>
<td>27.1±4.3</td>
<td>27.5±9.3</td>
<td>69</td>
<td>2.9</td>
<td>59±5.6</td>
<td>44.3±5.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.5%</td>
<td>0.5%</td>
<td>2.8±0.3</td>
<td>0.7±0.1</td>
<td>4.2±0.3</td>
<td>12.5±7.6</td>
<td>18.6±10</td>
<td>38</td>
<td>4.2</td>
<td>63.3±1.2</td>
<td>48.1±5.6</td>
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<tr>
<td></td>
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<td>1.0%</td>
<td>1.0%</td>
<td>4.5±0.8</td>
<td>1.2±0.3</td>
<td>6.9±1.4</td>
<td>13±7.0</td>
<td>9.9±4.8</td>
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<td>7</td>
<td>63±1.9</td>
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<td>1.5%</td>
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<td>8.3±0.7</td>
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<td>61.5±4.6</td>
<td>60.3±3.2</td>
</tr>
<tr>
<td>2</td>
<td>P: FRGC-FA/S</td>
<td>2.0%</td>
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<td>3.0±0.2</td>
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<td>0.8±0.1</td>
<td>6.6±1.3</td>
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<td>80.5±3.1</td>
</tr>
<tr>
<td>3</td>
<td>M: FRGC-S</td>
<td>1.5%</td>
<td>0.5%</td>
<td>4.5±0.7</td>
<td>1.0±0.2</td>
<td>6.9±0.9</td>
<td>12.7±4.4</td>
<td>12.6±4.9</td>
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<td>4.1</td>
<td>62.5±2.6</td>
<td>64.8±3.1</td>
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<tr>
<td></td>
<td></td>
<td>1.5%</td>
<td>0.5%</td>
<td>4.3±1.5</td>
<td>1.1±0.6</td>
<td>7.3±0.9</td>
<td>17.7±5.2</td>
<td>19.3±8.7</td>
<td>47</td>
<td>4.1</td>
<td>64.7±1.2</td>
<td>69.4±4.3</td>
</tr>
</tbody>
</table>

Note: The numbers indicate Average ± Standard Deviation

5.1 Compressive strength and elastic modulus

Generally, as reported in Table 4, the cylinders’ compressive strengths ($f_{cm}$) were 1% to 10% higher compared to the cubes’ compressive strengths ($f_{cu}$). However, some composites showed a contrasting trend where the compressive strength of the cubes was up to 25% greater relative to that of the cylinders; thus, now general conclusion could be drawn. It is good to highlight that the compressive strength and elastic modulus results of the non-fibrous slag-based cylinders (i.e. paste and mortar) were not provided in Table 4. It was challenging to test such matrices since the cast samples were highly brittle, and most of the samples were already broken before testing due to the rapid volume change caused by the high content of slag. On the other hand, the blended-based geopolymer matrix achieved cylinders’ compressive strength and elastic modulus of 56 MPa and 25 GPa, respectively. The sand addition occasioned 20% and 8% increases in both cylinders’ compressive strength (i.e. from 56 MPa to 67 MPa) and elastic modulus (i.e. from 25 GPa to 27 GPa) of the blended geopolymer matrix, respectively. Such observation was in consistency with previous research [16].

Fig. 6 shows the relationship between the cylinders’ compressive strength and the hybrid combinations for all the FRGCs achieved in this study. As shown in Fig. 6 and reported in Table 4, the cylinders’ compressive strength of the FA/S composites ranged from 61 to 82 MPa depending on the hybrid combination incorporated in the matrix, while the slag composites attained cylinders’ compressive strength ranging from 44 to 74 MPa. Thus, it was found that the incorporation of FA in the slag-based geopolymer matrix improved the compressive strength of the P:FRGC-FA/S by 6% to 38% relative to the P:FRGC-S composites. This could be due to the high volume change and drying shrinkage rate of the slag-based geopolymer matrix relative to that of FA/S geopolymer which might degrade the compressive
strength of the FRGC [42, 43]. Further, it was observed that the compressive strength of the FRGCs was directly related to the ST fiber volume fraction included in the composite which is supported by previous research results [16, 37]. The inclusion of fibers in the FA/S geopolymer matrix seemed to improve the cylinders’ compressive strength since all the P:FRGC-FA/S composites showed a higher compressive strength relative to their corresponding matrix (i.e. P:FRGC-FA/S matrix). On the other hand, it is worth mentioning that the mono-ST composites achieved a relatively lower compressive strength relative to the 0.5%PE+1.5%ST composites which might be due to the ST fiber settlement during the compaction process of the mono-ST composites. A similar observation was reported in previous research related to ECC [37]. Such a phenomenon was more severe in the slag-based geopolymer compared to FA/S geopolymer since the slag-based geopolymer showed weaker thixotropic behavior (i.e. less viscous) relative to that of FA/S geopolymer which might cause uncontrollable adverse fiber segregation.

Failure cracks

Fig. 6 – 28-days cylinders’ compressive strength versus PE and ST fiber volume fractions

Fig. 7 plots the elastic modulus results for the achieved FRGCs in this study. Clearly, the elastic modulus exhibited an opposite trend to that observed in the compressive strength (i.e. refer to Fig. 6). In other words, the elastic modulus of FA/S FRGCs was lower than that of slag-based FRGCs although the FA/S FRGCs achieved relatively larger compressive strength compared to the slag-based FRGCs. According to previous research [44], the elastic modulus of C-A-S-H gels in the slag-based geopolymer is higher than that of N-A-S-H gels in the FA-based geopolymer. The elastic modulus of P:FRGC-S composites was in the range of 26-30 GPa, while P:FRGC-FA/S composites had an elastic modulus ranging from 23-27 GPa depending on the fiber combination included in the matrix. The general trend observed in Fig. 7 was the more ST fibers included in the matrix, the higher the composite elastic modulus will be, to a limited extent. Further, the fiber addition improved the elastic modulus of the corresponding matrix regardless of the fiber type (i.e. P:FRGC-FA/S composites showed higher $E$ compared to P:FRGC-FA/S matrix, except the 0.5%PE+1.5%ST). It’s good to mention that the experimental values of elastic modulus observed in this study were significantly larger than their corresponding theoretical values (i.e. calculated based on the effective crack model theory [45]) reported in previous research [16, 21, 25]. Further, all the FRGC cylinders maintained their shape after the compression test (refer to Fig. 8) due to the PE fiber bridging effect [12, 16].

Fig. 7 – Elastic modulus of FRGC

Fig. 9 indicates the effect of sand addition on the compressive strength and elastic modulus of 1.5%PE+0.5%ST composite when included in both blended-based and slag-based FRGC. Clearly, the sand addition improved both compressive strength and elastic modulus of both FA/S and slag-based 1.5%PE+0.5%ST FRGCs. The improvements of the matrices compressive strength after sand addition were 4% and 35% for FA/S and slag-based FRGCs, respectively.

Fig. 8 – FRGC cylinders after compression test
Besides, the elastic modulus was enhanced by 19% when the sand was incorporated in both FA/S and slag-based 1.5%PE+0.5%ST FRGCs. According to previous studies [16, 26], sand addition improved the fracture properties of the geopolymer matrix significantly and thus increased their theoretical E.

![Image](57x494 to 284x684)

**Fig. 9 – Effect of sand addition on compressive strength and elastic modulus of 1.5% PE and 0.5% ST composite**

### 5.2 Flexural response

#### 5.2.1 Cracking and ultimate strengths

Figs. 10 a and b plot the flexural responses of both slag and blended FRGCs respectively. It is good to mention that the responses in Fig. 10 are the average of 4 specimens for each group at which the average curve presents the lowest deflection value in each group. Generally, the flexural responses of hybrid FRGCs laid between the envelope of mono FRGCs at which similar observations were reported for the tensile response in a previous study of the authors [16]. It is good to mention that the mono-PE FRGC attained a relatively nearby ultimate flexural strength to that of mono-ST FRGC when the binder was 100% slag (refer to Fig. 10a), while the ultimate flexural strength of the mono-PE blended-based FRGC outperformed that of mono-ST FRGC of the same matrix. Such observations might raise some doubts regarding the effectiveness of the hybridization concept in geopolymer composites relative to the cementitious one (FRGCs) wherein the effect of such a concept was more noticeable [46]. This could be due to the better interfacial strength between ST fiber and the cementitious matrix relative to that of geopolymer one [18]. However, it was challenging to capture the general trend following the average responses shown in Fig. 10, thus the flexural cracking and ultimate strengths reported in Table 4 are plotted in Fig. 11 for better comparison. In general, it was found that the slag-based FRGCs attained a relatively higher flexural cracking strengths in reference with the blended-based FRGCs (i.e. for 2% and 1.5% PE blended composites showed slightly higher cracking strength relative to slag composites). Such observation was identical to that of the tensile cracking strengths reported in previous research [16] at which the slag-based FRGCs showed higher cracking strengths relative to blended-based FRGC except for the composites including a higher fraction of PE fibers (i.e. PE volume ≥ 1.5%). Further, the flexural cracking strength of the slag-based FRGCs was directly related to the ST volume included in the composite which is in consistence with previous research [16]. On the other hand, the hybrid combinations did not show any effect on the flexural cracking strength of the blended-based FRGCs unlike the direct tensile cracking strength of the same composites reported in [16]. The reason for such behavior could be related to the fracture toughness of the slag- and blended-based geopolymer matrices reported in the previous study of the authors [16]. The higher toughness of the blended-based geopolymer matrix relative to the slag-based one (i.e. $K_{tip}$ and $J_{tip}$ as reported in [16]) could justify why the former had a higher cracking strength compared for the latter for composites with PE volume ≥ 1.5%. However, the use of higher content of ST (i.e ST volume ≥ 1%) has compensated the lower fracture toughness of the slag-based geopolymer matrix and therefore the slag-based FRGC attained a higher flexural cracking strength compared to blended-based FRGC when ST volume was ≥ 1%.

Additionally, the ultimate flexural strengths showed a similar trend to that of the flexural cracking strengths. In general, the slag-based FRGCs outperformed the blended-based FRGCs in terms of the ultimate flexural strength (i.e. except the 1.5%PE+0.5%ST composite was off this trend). The reason for such behavior could be due to the more compacted geopolymer matrix of slag-based FRGC compared to that of blended-based FRGC. As a result, the fiber-matrix bond of the slag-based FRGC would be higher than that of the blended-based FRGC, which contributed to the higher flexural and tensile strength of slag-based FRGC [16]. Further, the ultimate flexural strength of the slag-based FRGCs was dependent on the ST volume included in the matrix to a limited extent; however, the mono-PE composite showed a comparable ultimate flexural strength to that of the high-volume ST composite (i.e. ST≥1.5) in case of slag-based geopolymer matrix. Therefore, the hybrid combinations showed a poor performance in the slag-based geopolymers in terms of the ultimate flexural strength. On the other hand, such hybrid combinations showed a negligible effect on the flexural ultimate strength of the blended-
based FRGCs. It is worth highlighting that the observed behavior of the flexural response was unlike that of the direct tensile response reported in Alrefaei and Dai [16]. In other words, the flexural response of slag-based FRGCs was relatively alike the direct tensile response of blended-based FRGCs and vice versa (refer to Fig. 11 in [16]).

![Deflection hardening behavior of the hybrid composites](image)

**Fig. 10 – Deflection hardening behavior of the hybrid composites: a) slag FRGC b) blended FRGC**

![Cracking and ultimate flexural stresses versus PE and ST fiber volume fractions](image)

**Fig. 11 – Cracking and ultimate flexural stresses versus PE and ST fiber volume fractions**

**5.2.2 Deflections and ductility indices**

Fig. 12 illustrates the relationship between the deflection at ultimate load versus ST and PE fiber volume fractions for both slag and blended FRGCs. It was found that the PE volume fraction greatly influenced the deflection capacity of FRGC composites at ultimate flexural strength which is in agreement with previous studies [46, 47]. In addition, it was observed that the slag-based FRGCs exhibited relatively larger deflections at ultimate relative to the blended-based FRGCs (i.e. except 1.5%PE+0.5%ST composite). For example, the mono-PE slag-based composite achieved a deflection of 27 mm at the ultimate load, which is 22% higher than that of the mono-PE blended-based composite (i.e. 22 mm). Such observations were consistent with previous research findings [16] (i.e. refer to Fig. 13 in [16]). This could be due to the lower cracking strength of the slag-based FRGCs relative to blended-based FRGCs when the mono-PE combination was included in the matrix, which is more favorable for the pseudo strain hardening behavior [16].

![Deflection at ultimate load versus PE and ST fiber volume fractions](image)

**Fig. 12 – Deflection at ultimate load versus PE and ST fiber volume fractions**

However, for better understanding the effect of hybridization on the ductility of the FRGCs, Fig. 13 plots the calculated ductility indices (using equation 2 in section 3.2) versus the ST and PE volume fractions. Clearly, the ductility index of the composites was also directly related to the PE volume included in the FRGCs. In other words, the higher the PE volume in the composite, the more ductile (i.e. higher ductility index) the composite will be. However, it was challenging to decide which geopolymer matrix had a better ductility when incorporated with the hybrid combinations since the two curves in Fig. 13 almost intersect at the point of 1% PE. In general, the blended-based geopolymer matrix showed a higher DI for PE≤1, while the slag-based geopolymer matrix was more ductile for PE>1. Therefore, no clear conclusion could be drawn.
5.3 Effect of sand addition

Fig. 14 displays the effect of 212 µm sand addition on the deflection hardening behavior of the hybrid composite PE 1.5% and ST 0.5% when incorporated in both slag-based and blended-based geopolymer matrices. As shown in Fig. 14, the addition of sand was found to increase both cracking and ultimate flexural strengths of both slag-based and blended-based FRGCs. A similar observation was reported in both cementitious composites (ECCs) [47] and geopolymer composites [16, 26]. However, in order to summarize the effect of sand addition on the behavior of FRGCs, the average values reported in Table 4 are plotted in Fig. 15. As previously mentioned, the sand addition improved the flexural cracking strength of both blended- and slag-based FRGCs by 34% and 60%, respectively, while the ultimate flexural strength was enhanced by 6% and 64% for both blended- and slag-based FRGCs, respectively, in reference with their corresponding paste composites. On the other hand, the sand addition unexpectedly improved the ductility and deflection capacity of both blended- and slag-based FRGCs. However, the deflection improvement after sand addition was more noticeable in the blended-based FRGC (i.e. 59% increase in the deflection relative to the corresponding paste) compared to the slag-based FRGC (i.e. almost no change observed in the deflection capacity relative to the corresponding paste). Such improvement in the deflection behavior could be explained according to Nematollahi, et al. [26] research at which the use of appropriate content of fine sand (i.e. 212 µm) was found to maintain a desirable strain hardening behavior of FRGCs. Furthermore, the addition of sand has reduced the high volume change and drying shrinkage rate of the geopolymer paste matrices which might be the reason why the sand addition has improved the deflection behavior of geopolymer composites. Similar behavior can be observed in UHP-ECC at which the fine sand is used to minimize the shrinkage of the cementitious matrix [3]. However, such observation contradicted with the general trend of previous research wherein the sand addition adversely affects the strain (i.e. deflection) hardening behavior of composites due to the detrimental increase in the matrix fracture toughness that violates the pseudo strain hardening conditions [37, 48].

Fig. 15 – Summary of 212 µm sand addition effect on the flexural response of the hybrid composite PE 1.5% and ST 0.5% with different FRGC matrices

5.4 Cracking behavior

Fig. 16 shows the schematic representations of all FRGCs’ final conditions (i.e. over the full length of the specimen 300 mm) after testing wherein the numbers of the cracks passing the imaginary center line in each specimen are reported in the square brackets. It is good to mention that the reported cracked samples shown in Fig. 16 are based on the most cracked specimen from each group. All the cracks were marked using permanent marker after reaching the ultimate load and unloading the specimens; therefore, the actual number of cracks might be higher than the reported one since many micro-cracks were closed after unloading the specimens which made it difficult to trace all the cracks [49, 50].
After Sand Addition

Fig. 16 – Deflection cracking behavior of hybrid fiber FRGCs (The number shown in square brackets [ ] represents the number of the cracks).

Generally, the multiple-cracking behavior of the FRGCs was significantly reliable on the PE volume fraction incorporated in the composites. In other words, the higher the PE volume included in the matrix, the more saturated the multiple-cracking behavior will be. Such observation was previously recorded in both ECCs [37, 46, 47] and EGCs [16, 25]. As shown in Fig. 16, it was observed that the absence of PE fibers resulted in a highly brittle flexural failure with a single major crack (i.e. mono-ST FRGCs). Further, the number of cracks achieved by the slag-based FRGCs was relatively higher than that presented by the blended-based FRGCs. On the other hand, the multiple-cracking behavior of the 1.5%PE+0.5%ST composite was relatively enhanced after the addition of the sand to both slag- and
blended-based FRGCs. This was in consistence with the observation reported by Nematollahi, et al. [26] wherein the use relatively low-volume small-size sand (i.e. 30% of 212 µm sand by weight of binder) was found to improve the cracking behavior in addition to elastic modulus, ultimate strength, and strain capacity. However, the bulk of previous research mentioned that the sand addition violates the pseudo strain hardening conditions which in turn adversely affects the cracking behavior of the composite [16, 37, 47, 48].

![Image](image-url)

Fig. 17 – Average flexural crack spacing for all FRGCs

In order to better understand the flexural cracking behavior of FRGCs, the crack spacing \( S \) was calculated by dividing the distance between the farthest cracks over the number of cracks minus 1 \( (n-1) \) as mention in Alrefaei & Dai [16]. The calculated crack spacings are reported in Table 4 and presented in Fig. 17. As shown in Fig.17, the crack spacing dropped significantly by the addition of PE fibers to the geopolymer matrix. When the PE volume fraction was higher than 1%, the decrease of crack spacing was modest to a limited extent. This might confirm that the critical PE volume fraction was about 1% as reported by a previous study [18]. In addition, the crack spacing was found to be lower in slag-based FRGCs relative to that of blended-based FRGCs which is expected as the number of cracks was comparatively higher in slag-based FRGCs in reference with the blended-based FRGCs. After sand addition, the crack spacing was identical in both slag- and blended-based FRGCs (i.e. 4.1 mm as reported in Table 4) which was lower than that of their corresponding paste composites (i.e. 4.2 and 5.9 mm for P:FRGC-S and P: FRGC-FA/S respectively). This confirmed that the mortar composites (i.e. with sand) maintained a comparable crack spacing with the paste composites (i.e. without sand).

6. Summary and Conclusions

This study reports the experimental results of elastic modulus in addition to the deflection hardening behavior of the ambient-cured one-part steel-polyethylene FRGCs. Based on the observed results, the following conclusions can be summarized:

1) The inclusion of fibers improved the elastic modulus of the FRGCs relative to their corresponding none-fibrous matrices. Such improvements in the elastic modulus of FRGCs were directly related to the ST volume fraction included in the composite.

2) The slag-based FRGCs showed a relatively higher elastic modulus although they exhibited lower cylinders’ compressive strength relative to the blended-based FRGCs.

3) Generally, the flexural behavior of the slag-based FRGCs (i.e. modulus of rupture, deflection capacity, and multiple-cracking behavior) was comparatively better than that of the blended-based FRGCs. Whereas the deflection hardening behavior of both slag- and blended-based FRGCs was directly correlated to PE volume content included in the matrix, the ST content showed minor effects on the flexural cracking and ultimate strengths of the FRGCs.

4) The mono-PE FRGCs showed a comparative modulus of rupture relative to that of mono-ST FRGCs for both blended- and slag-based composites.

5) The use of low content (i.e. 30% by the mass of binder) of 212 µm sand improved the flexural response of both blended- and slag-based FRGCs without violating the deflection hardening conditions.

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Notations

The following symbols are used in this paper:

- \( S \): Crack spacing
- \( V \): Volume fraction
- \( M \): Mono
- \( P \): Polyethylene
- \( FA \): Fly Ash
- \( ST \): Steel Fiber
- \( PE \): Polyethylene Fiber
- \( FRGC \): Fiber Reinforced Geopolymer Concrete
- \( FA/S \): Fly Ash/Sand
- \( P:FRGC-FA/S \): Polyethylene: Fiber Reinforced Geopolymer Concrete (Fly Ash/Sand)
- \( M:FRGC-FA/S \): Mono: Fiber Reinforced Geopolymer Concrete (Fly Ash/Sand)
- \( M:FRGC-FA \): Mono: Fiber Reinforced Geopolymer Concrete (Fly Ash)
- \( P:FRGC-FA \): Polyethylene: Fiber Reinforced Geopolymer Concrete (Fly Ash)

Notations

The following symbols are used in this paper:
DI = ductility index;
E = composite elastic modulus;
Em = matrix elastic modulus;
f_cu = cube compressive strength of composite;
f_c = cylinder compressive strength of composite;
f_cm = cylinder compressive strength of matrix;
n = number of cracks;
\( \delta \) = crack spacing;
s_{LOF} = flexural cracking strength;
s_{MOR} = flexural ultimate strength;
\( \delta_{LOF} \) = mid-span deflection at cracking load; and
\( \delta_{MOR} \) = mid-span deflection at ultimate load.

References


