Optimized mix design for 180 MPa ultra-high-strength concrete

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\begin{abstract}
This study aims to optimize the proportion and constituents of the mixture used for fabricating 180 MPa ultra-high-strength concrete (UHSC) including coarse aggregate. Various parameters, such as the supplementary cementitious material (SCM) type and amount, water-binder (W/B) ratio, fine and coarse aggregate type and amount, chemical admixture, and mixing process, were considered and their effect on the compressive strength was examined. Based on several experimental tests, the optimized SCM proportions for cement, silica fume (SF), ground-granulated blast furnace slag (BFS), and gypsum are 55%, 20%, 20%, and 5%, respectively. The most appropriate W/B ratio, unit water content, and sand-total aggregate (S/a) ratio are 12.5%, 150 kg/m\textsuperscript{3}, and 0.35, respectively. The use of very fine SF with a specific surface area of 13.7 m\textsuperscript{2}/g and diorite, which is a type of coarse aggregate, improves the compressive strength of UHSC. The optimum amount of superplasticizer ranges between 1.3% and 1.6% and the most suitable slump flow is 750 mm. Finally, for this type of flowable UHSC, a mixing time and speed of 5 min and 40 rpm, respectively, and natural self-compacting without using a tamping bar are recommended. Based on the optimized mix design, UHSC with a compressive strength of 180 MPa can be certainly fabricated.

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\end{abstract}

1. Introduction

A recent trend in architectural engineering is to fabricate bigger and safer structures using high-performance construction materials. Various types of skyscrapers have been constructed worldwide as landmarks of cities such as the Burj Khalifa, Shanghai Tower, Lotte World Tower, and One World Trade Center.
Center. One of the key design and construction technologies of such skyscrapers is to effectively pump fresh concrete from the ground to the top of the structure and achieve structural integrity under very high longitudinal and lateral loads such as those caused by self-weight, wind, and earthquakes. These fundamental requirements on skyscraper construction can be met by developing self-consolidating ultra-high-strength concrete (UHSC). If a proper viscosity of the self-consolidating concrete is obtained by using high amounts of binder and superplasticizer, some of the segregation occurring during the pumping can be prevented with the smooth movement of fresh concrete in the pumping pipe [1]. Based on the use of UHSC with a compressive strength above 180 MPa for the fabrication, especially for columns, the very high longitudinal force due to the self-weight of the structures can also be resisted with enough structural integrity.

Several previous studies [2–7] were conducted to evaluate various material and structural performances of UHSC elements. Kimura et al. [2] examined the seismic behavior of UHSC columns with a compressive strength of about 200 MPa. They [2] used a cement premixed with silica fume (SF), sand, small-sized coarse aggregate with a maximum size of 13 mm, and a very low water-binder (W/B) ratio of 0.13 for the UHSC mixture. Based on experimental test results, it was reported that hooked-end steel fibers used as additional reinforcement enhance various structural properties, such as the flexural strength and the axial load carrying capacity up to a drift angle of 3%, and lead to low column damage with better crack dispersion. Maruyama et al. [3] tested full-scale UHSC columns in both summer and winter seasons to evaluate the stress distribution and crack patterns and reported several cracks around the steel reinforcements and the columns, which were generated by hydration heat and autogenous shrinkage. They [3] used cement premixed with 10% SF, a special crushed coarse aggregate from Morioka, Japan, superplasticizer, and a low W/B ratio of 0.15 to fabricate a self-consolidating UHSC mixture. The UHSC mixture achieved a slump flow value of 650 ± 100 mm. Yoo et al. [8] conducted autogenous shrinkage tests of UHSC and reported that its early age autogenous shrinkage increases with decreasing W/B ratio and supplementary cementitious materials (SCMs), i.e., fly ash and blast furnace slag, reduce the autogenous shrinkage of UHSC as the W/B ratio less than 0.2. Allena and Newton [9] noted that early age shrinkage of UHSC at 24 h was found to be 1758 μεε, about 58.5% of its ultimate shrinkage, meaning that a large portion of shrinkage strain of UHSC is developed at very early age. Czwirzen et al. [5] estimated the salt freeze–thaw resistance of UHSC with coarse granite and diabase aggregates, cement with amorphous SF (25% of the cement weight), quartz filler, short steel fibers, and low W/B ratios from 0.22 to 0.26. The heat curing, existence of coarse aggregates, and steel fibers were analyzed. They [5] reported that the lowest surface scaling and internal damage by freeze–thaw cycles were obtained using unheated UHSC with coarse aggregates. The inclusion of steel fibers had positive and negative effects on the internal damage and surface scaling, respectively. Bindiganaville [4] fabricated UHSC using cement, SF, quartz sand, a high-range water reducing agent, and high volume of steel fibers (6%) and tested it under impact-loading conditions. In their study, UHSC with steel fibers exhibited a superior impact resistance and an energy absorption capacity, three times greater than that of conventional concretes with compressive strengths of about 40 MPa and steel and polymeric fibers. To evaluate the fire resistance of UHSC, Lee et al. [6] used two different SCMs, that is, SF and ground-granulated blast furnace slag, fine and coarse aggregate, anhydrous gypsum to delay setting time, and a low W/B ratio of 0.125 for the UHSC fabrication. The compressive strength of 180 MPa UHSC deteriorated steeply with increasing temperature and failed due to thermal deformation at 300 ºC. Shin et al. [7] used the similar UHSC mixture to perform structural tests of reinforced UHSC columns with a compressive strength above 180 MPa and recommended to use high-strength steel reinforcing bars for UHSC columns in order to improve the strength and toughness.

Likewise, several studies [2–7] have been conducted to determine the mechanical and structural behaviors of UHSC. However, research regarding the detailed development process of such UHSC mixtures considering various factors, e.g., workability, strength characteristics, economic feasibility, environmental friendly through the use of SCMs, etc., is still very limited. Furthermore, since the mechanical properties of UHSC are highly sensitive to various factors, such as the ingredients, W/B ratio, and mixing and curing regimes, an optimized mix design stably providing such a very high compressive strength of 180 MPa needs to be established by considering extensive test variables and influential factors. Accordingly, in this study, various influential factors, i.e., the type and amount of SCM and aggregates, W/B ratio, chemical admixture, and mixing process, were considered to develop an optimized mix for UHSC with a compressive strength of 180 MPa and self-consolidating characteristics. From extensive experimental tests, an optimized flowable UHSC mixture, appropriate for skyscrapers, was suggested.

### Table 1 – Chemical compositions of cement.

<table>
<thead>
<tr>
<th></th>
<th>Normal cement</th>
<th>Specially fabricated cement</th>
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</thead>
<tbody>
<tr>
<td>LOI</td>
<td>0.65</td>
<td>0.85</td>
</tr>
<tr>
<td>SiO₂</td>
<td>21.25</td>
<td>21.80</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>5.15</td>
<td>4.85</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>3.45</td>
<td>3.63</td>
</tr>
<tr>
<td>CaO</td>
<td>64.15</td>
<td>63.60</td>
</tr>
<tr>
<td>MgO</td>
<td>2.15</td>
<td>2.00</td>
</tr>
<tr>
<td>SO₃</td>
<td>2.00</td>
<td>2.05</td>
</tr>
<tr>
<td>K₂O</td>
<td>1.25</td>
<td>1.00</td>
</tr>
</tbody>
</table>

a Normal Type I Portland cement.

b Specially fabricated cement for making UHSC.

### 2. Ingredients of UHSC composition

#### 2.1. Cement

To fabricate an 180 MPa UHSC mixture, the type of cement was first optimized. The chemical and mineral compositions of regular Type I Portland cement and specially produced UHSC cement are summarized in Tables 1 and 2. Since the cement is obtained from earth material, i.e., limestone, it is composed of numerous chemical compositions. Thus, only major
chemical compositions are given in Table 1. First, the amount of tricalcium aluminate (C₃A) was reduced to generate hardened, denser concrete with a lower W/B ratio. The amount of C₃A for the UHSC cement was reduced by 2% compared with that of regular cement (Table 2). In addition, the fineness (blaine) was lowered from 3300 to 3100 cm²/g to reduce the specific surface area and improve the filling factor during the fabrication of UHSC cement (Table 2). To reduce the soluble gypsum content, that is, the production of hemihydrate gypsum, the temperature of the outflow of the cement production mill was decreased from 105 °C to 90 °C. To improve the long-term hydration and densified microstructures, the amount of alite (C₃S) was reduced while the amount of belite (C₂S) was increased (Table 2). The C₂S augments the viscosity and fluidity in conventional concrete. However, in the case of the UHSC mixture, the increased amount of powder due to the addition of SF and ground-granulated blast-furnace slag (BFS) and the addition of superplasticizer (SP) lead to increased viscosity and fluidity. Thus, the amount of C₂S was lowered. On the other hand, the amount of C₃S was increased because it supports the production of calcium silicate hydrate (C-S-H) and enhances the long-term properties. Cement clinkers change the C₂S mineral depending on the plastic temperature. In general, C₃S, generated at high temperature, has a higher long-term strength than that generated at a lower temperature. Therefore, the clinkers for the UHSC cement were produced at high temperature and cooled rapidly. A polarizing microscope was used to determine the size and shape of the clinkers. The size of the C₂S in the clinker correlates with the rate of the temperature increase. As the size of C₂S decreases, the rate of the temperature increase rises.

### 2.2. Supplementary cementitious materials (SF and BFS)

The SF, which is necessary for the production of UHSC, improves the strength and reduces the viscosity of the cement. It is composed of condensed SiO₂ gas from the production of ferrosilicon and metal silicon used as deoxidizers. The particles are round and have an average size of 0.2–0.5 μm and specific surface area of 200,000 cm²/g; they can be classified as super fine-grained. The major roles of the SF in the mixture of UHSC are discussed below.

- Micro-filler effect: Particles with an average size of 0.2 μm fill the gaps between the water and cement particles in fresh concrete. In hardened concrete, the SF improves the strength and durability by filling voids made by hydrates.
- Pozzolanic reactivity: The Pozzolanic reaction is a chemical reaction between silica components and calcium hydroxide producing C-S-H. The rate of the reaction is about 40% after 3 days and 60% after 28 days when 10% are replaced with SF.
- Densified matrix: The fine pores are reduced when SF is added. The produced C-S-H has a low CaO/SiO₂ ratio of about 1.2–1.4, which contributes to the strength development by making the matrix structure dense.
- Reduced viscosity: The viscosity likely decreases when SF is added.

The workability of the cement paste increases with a better SF distribution. This eventually leads to an improvement in the fluidity of the concrete because SF with a good dispersibility can become more dispersible in the form of particles by external force of mixer and exert a ball-bearing effect due to the round shape of particles. The SF that is distributed in the form of particles improves the filling effect and replaces the water between the particles. This results in the production of redundant water and improves the fluidity. If the cement and SF are not mixed well, the fluidity of the fresh concrete deteriorates. Therefore, the cement and SF were premixed to overcome this drawback.

To enhance the strength and fluidity, SF with more than 92% SiO₂ and a fineness of 100,000–200,000 cm²/g was used. The effect is more prominent when the silica content is increased because the silica reacts with cement hydrates to produce C-S-H gel that fills the hydrate pores and thereby increases the compressive strength. Thus, SF with more than 92% SiO₂ should be used to fabricate UHSC. When using SF with a low fineness (100,000 cm²/g) and large particle size, the mixing time and viscosity significantly decrease, while the fluidity increases compared with that with a high fineness (200,000 cm²/g). Therefore, SF with a low fineness contributes to a better distribution of the entire binder mix in concrete with a compressive strength of 150–200 MPa and a W/B ratio below 15%. The SF used in UHSC should satisfy the quality requirements according to KS F 2567 [10] and have good distribution properties and round particles. Table 3 shows the results of the measurement of the specific surface area and SEM photographs of the SF types that were used.

In addition, to improve the packing density and fabricate eco-friendly UHSC, type II BFS with a specific gravity of 2.91 and a fineness ranging from 6000 to 8000 cm²/g was adopted. It was also expected that the addition of BFS improves the long-term strength of the UHSC.

### 2.3. Aggregates

To fabricate UHSC, the concrete aggregates must meet the following requirements: (1) a higher strength than the matrix strength, (2) similar specific gravity to that of the matrix, (3) durable under climate and utilization conditions, (4) an appropriate shape and grain size distribution to form UHSC with high fluidity and density, (5) absence of materials that

<table>
<thead>
<tr>
<th>Table 2 – Mineral compositions and physical properties of cement.</th>
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<tr>
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<td></td>
</tr>
<tr>
<td>C₃S</td>
</tr>
<tr>
<td>C₂S</td>
</tr>
<tr>
<td>C₂A</td>
</tr>
<tr>
<td>C₃AF</td>
</tr>
<tr>
<td>F-CaO</td>
</tr>
<tr>
<td>F-MgO</td>
</tr>
<tr>
<td>Gypsum</td>
</tr>
<tr>
<td>K₂SO₄</td>
</tr>
<tr>
<td>Blaine (cm²/g)</td>
</tr>
</tbody>
</table>

* Normal Type I Portland cement
b Specially fabricated cement for making UHSC
Table 3 – Specific surface area and SEM images for different types of SF.

<table>
<thead>
<tr>
<th>Type</th>
<th>Specific surface area (m²/g)</th>
<th>SEM image 10,000×</th>
<th>SEM image 30,000×</th>
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<tbody>
<tr>
<td>SF-A</td>
<td>13.7</td>
<td><img src="image1" alt="SEM image" /></td>
<td><img src="image2" alt="SEM image" /></td>
</tr>
<tr>
<td>SF-B</td>
<td>11.9</td>
<td><img src="image3" alt="SEM image" /></td>
<td><img src="image4" alt="SEM image" /></td>
</tr>
<tr>
<td>SF-C</td>
<td>14.4</td>
<td><img src="image5" alt="SEM image" /></td>
<td><img src="image6" alt="SEM image" /></td>
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<tr>
<td>SF-D</td>
<td>10.9</td>
<td><img src="image7" alt="SEM image" /></td>
<td><img src="image8" alt="SEM image" /></td>
</tr>
</tbody>
</table>

potentially deteriorate the properties of concrete, and (6) a good fire resistance. The aggregates in UHSC are more important than in normal- and high-strength concretes. Thus, coarse aggregates with sizes below 10 mm and a strength higher than that of the matrix should be used for UHSC. Accordingly, coarse aggregates with a strength above 180 MPa, which is the target compressive strength, and a secured fire resistance up to 1200 °C were selected for this study. A nationwide aggregate investigation was conducted in South Korea and the aggregate properties were analyzed to identify aggregates satisfying the above-mentioned requirements.

Based on the measurements of the compressive strength, three optimized types of coarse aggregate for UHSC were selected, that is, diorite, shale, and andesitic rock with compressive strengths of about 301, 299, and 336 MPa, respectively, which are much greater than 200 MPa (Fig. 1). To assess the aggregate properties, X-ray fluorescence (XRF) analysis and alkali reaction and fire resistance tests were performed. The XRF results are summarized in Table 4. The diorite aggregate has the largest SiO₂ content, while it has the smallest Fe₂O₃ and MgO concentrations. The andesitic rock has the least amount of K₂O among the components with low melting points. The alkali reaction test was carried out in accordance with KS F 2545 [11]; the test results are shown in Table 5.

The hysteresis curve in an electronic furnace (Fig. 2) increases by 25 °C per min up to 1200 °C, where the temperature remains for 60 min before it decreases at a rate of 25 °C/min. The diorite remains almost the same during the 1200 °C heating test, which proves that it is most suitable for the use in UHSC, whereas the shale and andesitic rock aggregates have melting of the surface and found to have eluted elements. In addition, based on our preliminary compression tests on UHSCs with three different coarse aggregates [12], the one with diorite provides the highest compressive strengths both at early stages and over the long term. A W/B ratio of 0.11 and unit water content of 130 kg/cm³ were used...
for this preliminary test. The ratio of cement:SF:BFS:gypsum was 50:20:25:5 and the slump flow values ranged from 715 to 725 mm according to ASTM C1611 [13]. The highest compressive strengths of UHSC with diorite are about 201 MPa after 28 days under steam-curing conditions (80 °C) for 7 days.

Three different types of fine aggregates were adopted: washed sand, electric arc furnace (EAF) slag, and silica sand. Regular washed sand with a specific gravity of 2.65 and fineness modulus of 3.05 was adopted. The properties of the EAF slag and silica sand are the following: EAF slag with a specific gravity of 3.58 and fineness modulus of 3.06 and silica sand with a specific gravity of 2.67 and fineness modulus of 1.14.

### 2.4. Water-reducing admixture (superplasticizer)

A considerably large flow loss of the UHSC mixture causes problems at construction sites that require a long transfer of materials, especially for the pumping of skyscrapers. Polycarboxylic acid-based superplasticizer (SP), which recently was developed by Dongnam Co., Ltd, has made it possible (flowable and low flow loss) to produce UHSC with a compressive strength over 100 MPa. Thus, SP was adopted in this study to produce flowable UHSC. Its chemical characteristics are summarized in Table 6.

### 3. Test program

#### 3.1. Mixing process

A non-gravity mixer was used in this study to preblend the cementitious binders. The two axes of the mixer rotate at a steady speed rate powered by a chain actuator. When the raw materials are pushed up in the mixing chamber by rotating paddles, a non-gravity state is formed with an increase of effective volume where a weightless zone is created to complete mixing.
4.1. Effects of the gypsum concentration and fineness

The early-stage setting behavior of the cement paste mainly depends on the reaction of C_3A. If C_3A reacts with water, the cement paste immediately stiffens. Therefore, gypsum is added to retard this abrupt setting behavior [15,16]. Potgieter et al. [17] suggested the use of 3-5% (by weight) gypsum in the clinker. Because the addition of gypsum also leads to early-stage strength development and decreases the drying shrinkage of the hardened cement paste [18], a proper amount of gypsum needs to be determined for UHSC with a much higher strength than that of ordinary concretes. To determine the amount of gypsum appropriate for UHSC, an X-ray diffraction (XRD) analysis was carried out on UHSC matrix samples with a W/B ratio of 0.125. The changes of the anhydrite and ettringite peaks of UHSCs with different amounts of anhydrous gypsum and curing ages are shown in Figs. 3 and 4. Three different amounts of gypsum (0%, 5%, and 10% of the total amount of binder materials) were considered, while the SF and BFS concentrations were fixed to 20% of the total binder content. To determine the optimum amount of gypsum, a standard natural anhydrous gypsum with a specific gravity of 2.7 and fineness of 3500 mm²/g was used. The amount of cement was reduced from 60% to 50% by increasing the amount of gypsum. Without the addition of anhydrous gypsum, anhydrite and ettringite peaks were not observed (at all ages), while a clear anhydrite peak was detected for the samples with 5% and 10% gypsum (Fig. 3). With increasing curing age, the peak intensity of anhydrite reduces because it is replaced with ettringite during the hardening process [19]. Due to this replacement, the peak intensity of ettringite increases with the curing age when 5% and 10% gypsum are added (Fig. 4). The mixture without gypsum therefore does not show an ettringite peak (Fig. 4). A greater decrease of the anhydrite peak intensity was observed in the samples with 5% anhydrous gypsum as compared with its counterpart (10% gypsum), indicating that they could effectively delay the abrupt setting of the cement paste and develop an early-stage strength. Accordingly, similar to a previous study [17], the proper amount of anhydrous gypsum is 5% of the total amount of binder materials.

To examine the effect of the gypsum fineness on the compressive strength of UHSC, natural anhydrous gypsum was finely crushed to exhibit a fineness of 6000 and 8000 cm²/g. Thus, three finenesses of 3500, 6000, and 8000 cm²/g were considered along with the hybrid use of 6000 and 8000 cm²/g gypsum. Because the optimum amount of gypsum was determined to be 5%, the gypsum amount was fixed to 5% during the evaluation of the effect of its fineness on the compressive strength. Fig. 5 shows that the 91-day compressive strength slightly increases with increasing gypsum fineness. In addition, the highest compressive strength was obtained for the UHSC mixture with hybrid use of equal amounts (2.5%) of 6000 and 8000 cm²/g gypsum because finer gypsum effectively 

<table>
<thead>
<tr>
<th>Diorite</th>
<th>13.75</th>
<th>71.17</th>
<th>Harmless</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shale</td>
<td>18.59</td>
<td>96.52</td>
<td>Harmless</td>
</tr>
<tr>
<td>Andesite</td>
<td>14.74</td>
<td>78.53</td>
<td>Harmless</td>
</tr>
</tbody>
</table>

### Table 6 – Chemical characteristics of polycarboxylic acid-based superplasticizer for UHSC.

<table>
<thead>
<tr>
<th>Color</th>
<th>Specific Gravity</th>
<th>pH</th>
<th>Solids (%)</th>
<th>Viscosity (cps)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dark brown</td>
<td>1.10</td>
<td>7.40</td>
<td>30.1</td>
<td>58</td>
</tr>
</tbody>
</table>
Fig. 3 – Change in anhydrite according to amount of anhydrous gypsum: (a) 3 days, (b) 7 days, (c) 28 days, and (d) 91 days.

Fig. 4 – Change in ettringite according to amount of anhydrous gypsum: (a) 3 days, (b) 7 days, (c) 28 days, and (d) 91 days.
fills the spacing between coarser gypsum particles. Thus, the hybrid use of gypsum with finenesses of 6000 and 8000 cm$^2$/g is recommended to improve the mechanical strength of UHSC.

4.2. Effect of the W/B ratio

The porosities of the aggregate, cement paste, and interfacial transition zone (ITZ) are the most important factors that determine the strength of UHSC. The porosities of the cement paste and ITZ are affected by several parameters such as the composition, mix proportion, and compactness of the concrete. At an appropriate workability, the W/B ratio most significantly affects the porosities of the hydrated cement paste and ITZ. A lower W/B ratio is known to increase the strength of concrete and leads to a decrease in the total porosity and finer pore structure [20]. However, such a decrease of the W/B ratio results in difficulties during mixing, concrete casting, and concrete compacting due to the insufficient workability. Therefore, a binder mix with an appropriate construction ability that meets desired strength criteria is very important for the development of UHSC. Therefore, the effect of the W/B ratio on the compressive strength of UHSC after various curing days was examined. The proportions of the mixture and properties of fresh concrete are summarized in Table 7. The W/B ratio ranges from 11.5% to 14.5% and the portions of cement, SF, BFS, and gypsum are 55%, 20%, 20%, and 5%, respectively. The amount of SP increases with decreasing W/B ratio since the flowability is generally reduced with increasing binder material amount, relative to the amount of water. Thus, similar slump flow values ranging from 800 to 860 mm were obtained for all mixtures.

A summary of the compressive strength data of UHSCs with various W/B ratios is shown in Fig. 6 based on the curing age. At a very early-stage (3 days), the highest compressive strength was observed in the UHSC mixture with the highest W/B ratio of 14.5%; the magnitude decreased with decreasing W/B ratio because of the increased amount of SP, delayed setting time, and early-stage strength development [21]. On the other hand, similar compressive strengths were obtained between 7 and 28 days, regardless of the W/B ratio. After 28 days, a higher compressive strength was obtained at lower W/B ratios up to 12.5% because the lower W/B ratio leads to a smaller porosity of the cement paste [22]. Due to the smaller porosity, the UHSC mixture with the W/B ratio of 12.5% exhibits the highest 91-day compressive strength. Interestingly, the UHSC mixture with the lowest W/B ratio of 11.5% exhibits the smallest compressive strength due to the insufficient amount of water and incomplete hydration of the cement (not able to produce full strength).

In order to determine the relationship between the W/B ratio and compressive strength, the porosity and microhardness of the ITZ between the cement matrix and coarse aggregate were analyzed. To measure the porosity and microhardness of the ITZ, a cylindrical sample with a diameter of 100 mm and a height of 200 mm was prepared. From the center of the hardened sample, area where aggregate and matrix interface is clear and distinctive was cut into a prismatic piece with a dimension of $10 \times 10 \times 10$ mm. To stop the cement hydration, the cut-out piece was dried for 24 h using acetone. Subsequently, the acetone was removed using an aspirator for 30 min. The piece was then placed in a drier for 24 h at 40°C and afterwards stored in a vacuum desiccator. Mounting was carried out next. Low-viscous epoxy resins and a curing agent were used at a ratio of 5:1. The mounting solution was filled in a mold cup until the specimen was submerged. The cup was placed in a 40°C drier for 24 h to harden. A 1 mm cross section of the hardened specimen was cut using cutting equipment. Subsequently, SiC paper (#1000–2000) was used to remove scratches made by cutting the sample and to level the specimen. Furthermore, the specimen was polished using diamond suspension (6, 3, 1, and 0.25 μm). During polishing, the absence of scratches on the specimen and appropriate glossy surface were checked before continuing the polishing using.

| Table 7 – Mixture proportions and fresh properties of UHSCs with various W/B ratios. |
|---|---|---|---|---|---|---|
| W/B (%) | S/a | W (kg/m$^3$) | C:SF:BFS:Gy | SP (B × %) | S. flow (cm) | Air$^a$ (%) | T60–T50$^b$ (s) |
| 14.5 | 1.30 | 80/80 | 2.3 | 5.7 |
| 13.5 | 1.50 | 80/80 | 2.3 | 5.4 |
| 12.5 | 1.70 | 83/86 | 2.5 | 4.0 |
| 11.5 | 1.90 | 80/80 | 3.0 | 5.5 |

Note: W/B, water-to-binder ratio; S/a, sand to total aggregate ratio; W, unit water content; C, cement; SF, silica fume; BFS, ground granulated blast furnace slag; Gy, gypsum; SP, superplasticizer; B, binder; S. flow, slump flow.

$^a$ Air content

$^b$ T50, T60 = times that concrete requires to reach 500 and 600 mm of slump flow as per ASTM C1611 [11], so that T60–T50 indicates time difference between T60 and T50.
the suspension with the next smaller diamond particle size. In addition, the speed of the polishing equipment was set to 150rpm at a compressive load of 10 N. Finally, the polished specimen was analyzed with electron probe microanalysis (EPMA); magnified backscattered electron (BSE) images and the microhardness were obtained. The SHIMADZU-1610 and Mitutoyo HM-100 instrumentations were used to take the BSE images and evaluate the microhardness, respectively. The test details can be found elsewhere [12].

The effect of the W/B ratio on the porosity and microhardness based on the distance from the ITZ between coarse aggregate and matrix is shown in Fig. 7. The porosity near the ITZ is noticeably influenced by the W/B ratio. With decreasing W/B ratio to 12.5%, the porosity significantly reduces, whereas it slightly increases from a W/B ratio of 12.5–11.5%. This trend is identical with that obtained by compressive strength measurements. In addition, the UHSC mixture with a W/B ratio of 12.5% shows the highest microhardness near the ITZ (Fig. 7b), while those with W/B ratios of 11.5% and 14.5% have the smallest microhardness. Based on these observations, it can be concluded that the W/B ratio of 12.5% is the optimum value for the UHSC mixture; it maximizes its compressive strength. A higher porosity and smaller microhardness were obtained for the mixture with a W/B ratio of 11.5% because of the insufficient mixing of all powders due to the lack of water. The insufficient mixing results in an increased inhomogeneity.

4.3. Effect of the water content

To determine the optimum water content for the production of UHSC, three different unit water contents (140, 150, and 160 kg/m$^3$) were considered. The proportions of the binder materials and S/a ratio were identical to those given in Table 7 and a W/B ratio of 12.5% and 1.5% SP were applied to fabricate UHSC mixtures with slump flow values ranging from 720 to 750mm and air contents ranging from 2.6% to 3.0%. Fig. 8 shows the compressive strengths of the UHSCs mixtures with different water contents depending on the curing age. Although the compressive strength did not notably change depending on the water content, the mixtures with water contents of 150 and 160 kg/m$^3$ exhibit a slightly higher ultimate compressive strength after 91 days than the mixture with 140 kg/m$^3$ water. However, based on a previous study by Wiegrink et al. [23], higher water contents lead to higher weight loss with age and higher shrinkage. Thus, a water content of 150 kg/m$^3$ is determined to be optimal for the UHSC mixture.

4.4. Effects of the fine aggregate ratio (S/a) and type

To examine the effects of the fine aggregate type and ratio (S/a), three different types, that is, washed sand, EAF slag, and silica sand, and S/a ratios ranging from 0.14 to 0.45 were considered. The previously determined optimum W/B ratio,
water content, and gypsum amount (12.5%, 150 kg/m³, and 5%, respectively) were used to evaluate the effect of the fine aggregate type. When the S/a ratio increased, the slump flow decreased such that the SF amount increased with increasing S/a ratio, ranging from 1.4% to 1.7%. Thus, reasonable slump flow values ranging from 680 to 840 mm were obtained for all UHSC mixtures.

The compressive strength data for the UHSC mixtures with various fine aggregates and S/a ratios are summarized according to the curing age in Fig. 9. In the case of washed sand, the S/a ratios of 0.35 and 0.45 lead to slightly higher compressive strengths than that of 0.4. On the other hand, the other types of fine aggregate (i.e., EAF slag and silica sand) show a clear decrease in the compressive strength with increasing S/a ratio, as shown in Fig. 9b and c. In addition, the use of silica sand provides the highest compressive strength, followed by washed sand and EAF slag. It can be concluded that the compressive strength of UHSC slightly reduces with increasing S/a ratio, regardless of the fine aggregate type. Because the price of silica sand is higher and it is difficult to obtain silica sand in many countries, including South Korea, washed sand with an S/a ratio of 0.35 is proposed to be optimal for UHSC.

4.5. **Effect of the SF and BFS concentrations**

Because limestone, which is a main ingredient in Portland cement, is a finite resource, it is important to reduce its amount for the use in concrete fabrication. Therefore, to replace a portion of the cement with SCMs, SF and BFS were incorporated in this study. The optimum W/B ratio of 12.5%, water content of 150 kg/m³, and S/a ratio of 0.35 were adopted. To determine the optimum replacement ratios of SF and BFS, the compressive strengths of the UHSC mixtures with various replacement ratios of SF and BFS ranging from 15% to 30% of the total binder materials were measured and analyzed. In addition, in the case of SF, the effect of the replacement ratio on the fluidity and viscosity of the cement paste was also estimated according to ASTM C1437 [24] and ASTM C1749 [25], respectively. The mini slump and plastic viscosity values determined for cement paste with different replacing ratios of SF ranging from 0% to 30% are shown in Fig. 10. Note that the fluidity increases and the plastic viscosity decreases when the replacement ratio of SF increases up to 20%. However, beyond a ratio of 20%, the fluidity decreases while the plastic viscosity increases with increasing replacement ratio. Therefore, to fabricate a self-consolidating UHSC mixture, the optimum SF replacement ratio is about 20%.

The effect of the SF replacement ratio on the compressive strength is shown in Fig. 11a. The SF is known to be an excellent micro-filler that increases the concrete’s compressive strength and durability by filling pores based on pozzolanic and hydration reactions. However, in excess use, the amount of ultrafine particles increases, resulting in the deterioration of the workability and strength due to the increased amount of water required. Thus, the appropriate replacing ratio of SF is approximately 15–20% (Fig. 11a) because it provides a higher compressive strength than a larger replacement ratio of 25%. Consequently, based on the fluidity and compressive strength...
measurements, the optimum replacing ratio of SF is 20%. Bache [26] also developed very-high-strength concrete with a compressive strength in the range from 150 to 400 MPa, called compact reinforced composite (CRC), and its superb mechanical strength was achieved by incorporating high amounts of SF ranging from 20% to 25% and a low W/B ratio of 0.16 [27]. The slightly higher optimum SF contents for CRC might be caused by its higher W/B ratio, leading to a better workability, than that of UHSC, so that the optimum replacing ratio of SF, i.e., 20%, suggested in this study can be considered as suitable.

Fig. 11b shows the effect of the BFS replacement ratio on the compressive strengths of UHSCs. The 20% SF was used for the evaluation of the BFS effect. Fig. 11b shows that the highest compressive strength of UHSC is obtained when the replacement ratio of BFS is 20%, which is consistent with the findings of Shi et al. [28]. They [28] reported that the highest compressive strength of UHSC was achieved in a combination of SF and BFS when the content of BFS is ranged from 10% to 20%. This means that the compressive strength improves by increasing the amount of BFS from 15% to 20%, whereas the increase of the BFS decreases the compressive strength beyond a replacement ratio of 20%. Therefore, the optimum replacement ratio of BFS is considered to be 20%.

4.6. Effect of the high-range water-reducing admixture (SP)

To optimize the amount of SP regarding the compressive strength, various amounts of SP were applied, ranging from 1.3% to 2.1% (Fig. 12). The mix proportion used for this investigation is identical to that used in the previous cases, that is, a W/B ratio of 12.5%, water content of 150 kg/m³, S/a ratio of 0.35, and SCM ratios of 55:20:20:5(cement:SF:BFS:Gy). The compressive strength was measured after 3 and 56 days. Fig. 12 shows that the early-stage compressive strength notably reduces with increasing SP amount. This is attributed to the delayed stiffening development of the concrete with the higher SP amount, which is consistent with the findings of Pacheco-Torgal et al. [21] and Lemieux et al. [29].
of the concrete was also observed before demolding in accordance with the increase of the SP amount. Compared with the early-stage strength, the 56-day compressive strength of UHSC is insignificantly influenced by the amount of SP. However, the compressive strength measured after 58 days is slightly smaller when the amount of SP is higher than 1.7%. Therefore, an optimum SP amount ranging from 1.3% and 1.6% is recommended; the appropriate amount can be determined based on slump flow measurements within this range.

To determine the proper slump flow range, the relationship between the compressive strength and slump flow value is illustrated in Fig. 13. Identical mixture proportions were used, but the amount of SP was varied to obtain different slump flow values. It is interesting to note that the compressive strength of UHSC is influenced by the slump flow. Lemieux et al. [29] reported that normal-strength self-consolidating concrete with W/B ratios of 0.33 and 0.38 exhibits the highest compressive strength at a medium slump flow level ranging from 640 to 700 mm compared with other concretes (low level = 600–640 mm, high level = 700–735 mm). However, in the case of UHSC with a very low W/B of 12.5%, the ultimate compressive strength increases up to a slump flow value of about 750 mm and the highest compressive strength was maintained up to about 830 mm. Thereafter, the compressive strength significantly decreases. These inconsistent results might be attributed to the significantly different W/B ratios. Because the UHSC has a high amount of fine dry powders, its fresh mixture has a high viscosity and is very sticky, leading to the insufficient compaction of the mold. The imperfect filling and compaction of cylindrical molds lead to a decrease in the strength; the impact is significant in the case of UHSC compared with ordinary concretes. Therefore, a higher compressive strength of UHSC is obtained at a higher slump flow level (750–830 mm). Because the higher slump flow requires a higher amount of SP, the slump value of about 750 mm is recommended for UHSC based on economic aspects, which is included in a range of acceptable slump flow of self-consolidating concrete reported by El-Chabib and Nehdi [30]. Massana et al. [31] also reported that the slump flow ranging from 660 to 750 mm is suitable for many normal applications of self-consolidating concrete.

4.7. Effect of the coarse aggregate type

Based on preliminary tests using diorite as coarse aggregate, UHSC was determined. To compare its effectiveness with that of other types of coarse aggregate, three additional types of coarse aggregate (i.e., iron ore, EAF slag, and clinker) were considered. The mix proportions that were used were also identical with that used for the previous cases. The measured compressive strengths are summarized in Fig. 14. The diorite aggregate is denoted as crushed in Fig. 14. It is notable that iron ore and clinker are inappropriate for UHSC because they exhibit much smaller compressive strengths than the other aggregates (crushed and EAF slag aggregates). Due to the insufficient strength development, their compressive strengths were only measured up to 28 days. The EAF slag coarse aggregate has a higher strength than the iron ore and clinker but exhibits a smaller compressive strength than the crushed aggregate (diorite), which is inconsistent with the findings of Papayianni and Anastasiou [32]. In their study, the use of EAF slag aggregate increases the compressive strength of HSC (f′c of about 70 MPa) up to 20% compared with using normal crushed limestone aggregate. In accordance with a previous study by Manso et al. [33], the minimum compressive strength of the EAF slag aggregate is 130 MPa, which is much smaller than the compressive strengths of diorite and UHSC used in this study. Thus, the UHSC mixture with the EAF slag aggregate exhibits a lower compressive strength than that with crushed aggregate, which is due to the insufficient strength of the EAF slag.

4.8. Effect of the SF type

Five different types of SFs were considered in this study, as summarized in Table 3, and their impact on the compressive strength of UHSC with a mix proportion identical to that of the previous cases was analyzed (Fig. 15). The only difference between the SF types is the particle size. The use of SF-A with a specific surface area of 13.7 m²/g leads to the highest compressive strength, although the differences in the compressive strengths are rather minor. The other three types of SF (i.e.,
SF-B, -C, and -D) have similar compressive strengths, although they have different specific surface areas. Thus, the SF with a specific surface area of 13.7 m²/g is recommended for UHSC, but the impact of the SF size is relatively insignificant compared with other factors such as the W/B ratio, type of fine and coarse aggregates, amount of SCMs, and flowability.

4.9. Effects of the mixing time, speed, and compacting method

Based on several experimental tests, the optimized mix for UHSC was determined. Before applying it practically, the effects of various concrete mixing and casting processes on the compressive strength of UHSC were investigated. Fig. 16 shows the implications of the mixing time, speed, and compacting method on the compressive strength at various curing ages. As shown in Fig. 16a and b, although the difference in the compressive strengths depending on the mixing time and speed was insignificant, a mixing time of 5 min and speed of 50 rpm led to a slightly higher compressive strength than that used in other cases (10 min mixing at 40 rpm speed). It is interesting to note that the untamped concrete exhibits a higher compressive strength than the tamped concrete, which is contrary to ordinary concrete (Fig. 16c). This is because the UHSC mixture is a self-consolidating concrete with a very high slump flow and viscosity; the tamping rod rather tangles the fresh flowable concrete, causing poorer compactness, compared with the mixture that naturally fills the mould. Thus, natural self-compaction is recommended for the flowable UHSC mixture.

5. Conclusions

In order to optimize the proportions and composition of the mixture used for the fabrication of UHSC, various parameters were considered in this study and their effect on the compressive strength was investigated. Based on the above-mentioned test results, a UHSC mixture with a compressive strength of about 180 MPa was developed. The following conclusions can be drawn:

1) The peak intensity of anhydrite can be effectively reduced by including 5% gypsum, indicating the delay of the abrupt stiffening of cement paste and enough early-age strength development. The use of finer gypsum improves the strength of UHSC. Consequently, the inclusion of 5% gypsum with fineness of 6000 and 8000 cm²/g is recommended.

2) The decrease of the W/B ratio decreases the porosity and increases the microhardness near the ITZ and the ultimate compressive strength of UHSC up to 12.5%. However, beyond that, the porosity increases, while the microhardness and compressive strength decrease with decreasing W/B ratio. Therefore, an optimum W/B ratio of 12.5% is suggested for the fabrication of UHSC.

Fig. 15 – Effect of SF type on the compressive strength.

Fig. 16 – Effects of mixing time, speed, and compacting method on the compressive strength.
3) A water content of 150 kg/m³ and SF with a specific surface area of 13.7 m²/g are proposed to be the optimum values for UHSC. However, their effect on the compressive strength is relatively small compared with that of other factors.

4) The compressive strength of UHSC slightly decreases with increasing S/a ratio. The use of washed sand with an S/a ratio of 0.35 is recommended. In addition, using diorite as a coarse aggregate is most suitable for the fabrication of UHSC with a compressive strength above 180 MPa.

5) To achieve self-consolidating properties and a higher compressive strength, equal replacement ratios of SF and BFS of 20% are recommended. Therefore, the optimum proportions of cement, SF, BFS, and gypsum among the total binder materials are 55%, 20%, 20%, and 5%, respectively.

6) Based on the compressive strength measurements, the optimum range of the SP amount is 1.3–1.6%. In addition, the most suitable slump flow value of UHSC is about 750 mm based on economic aspects.

7) A mixing time and speed of 5 min and 40 rpm, respectively, along with natural self-compaction without an artificial tamping rod are suggested for the development of flowable UHSC.

Since the properties of fresh and hardened UHSC vary according to the physical properties and chemical compositions of ingredients used, the various methods suggested above to develop flowable UHSC with the compressive strength of 180 MPa or higher can be practically adopted only after checking the material properties using accessible ingredients.

Conflicts of interest

The authors declare no conflicts of interest.

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